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Solvay USA Inc. Houston Plant

CERTIFIED MAIL: Return Receipt Requested (7008 0150 0001 2472 2999)

RECEIVE

October 3, 2013

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733 **CCT 22 2013**

Air/Toxics & Inspection Coordination Branch 6EN-A

Re:

Solvay Benzene NESHAP, Subpart FF, Quarterly Report

July 1, 2013 to September 30, 2013

EPA ID No.: TXD008099079

Dear Mr. Robinson:

Solvay USA Inc. formally Rhodia Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Solvay receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Solvay submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Solvay USA Inc. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

• Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the average temperature of the gas stream in the combustion zone for the TS Vapor Combustor was <50°F below design temperature when being used as the control device for the TS storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Willia J. M. Coming

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

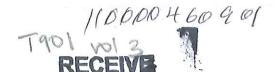
Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Solvay USA Inc. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: September 30, 2013

Inspection	Was inspection	Exceptions Noted
<u> </u>	performed?	* # 19 h
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		ļ

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.





6EN-A

Air/Toxics & Inspec Coordination Branch

July 26, 2013

Via FedEx

Chief, Environmental Enforcement Division **Environment and Natural Resources** Division U.S. Department of Justice Box 7611 Ben Franklin Station Washington. DC 20044-7611

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Jan Gerro U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6RCEA Dallas, TX 75202

David Schnare U.S. Environmental Protection Agency Headquarters Ariel Rios South Building, Rm #2117B 1200 Pennsylvania Avenue, N.W. Mailcode 2242A Washington D.C. 20460

Himanshu Vyas U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6ENAT Dallas, TX 75202

Re: Rhodia Inc. - Consent Decree Semi-Annual Report U.S. v. Rhodia, USDC (N.D. Ind.) Case No. 2: 07-CV-134-WCL

Ladies and Gentlemen:

In accordance with Section VII of the Consent Decree ("CD") entered in the above-entitled matter, enclosed please find the Semi-Annual Report for Rhodia's Houston, Texas facility. The Report satisfies, together with the other supporting documents enclosed, Rhodia's obligation to report on certain matters under the CD within 30 days after the end of each half calendar year (see CD ¶¶ 21-23).

If you have any questions or wish to discuss this submittal, please do not hesitate to contact me.

Thank you.

Sincerely yours

JSL:vkd Enclosure

F. Sardo Bcc:

Rhodia Inc. - Houston #8 and #2 Plants Consent Decree Semi-Annual Report for Period Covering January 1 to June 30, 2013 Civil Action No.: 2: 07-CV-134-WCL

- 1. Effective Dates:
 - a. Houston #8 July 1, 2009
 - b. Houston #2 April 1, 2014
- 2. Status of Construction or Compliance Measures Necessary to Meet Emissions Limits.

The plant has now completed the construction and implementation of all compliance measures necessary to meet the CD emission limits for #8 Unit. The SO₂ abatement was started up on November 19, 2008.

Construction has commenced for the Houston #2 SO₂ abatement system. The foundation has been poured, structural steel installed, the scrubber vessel has been set, and pumping and piping installation has commenced.

- 3. Compliance Issues and Proposed or Implemented Solutions
 - (a) Houston #8 Long-Term SO₂ Limit of 1.70 lbs/ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #8 unit operated below the permitted 1.70 lbs SO₂/ton of acid produced from July 1, 2012 to June 30, 2013.
 - (b) Houston #8 Short-term SO₂ limit of 3.00 lbs/ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.
 - (c) During the reporting period, the plant has not encountered any problems, and does not anticipate encountering any problems, with any of the conditions of the Consent Decree or any applicable permits or any other event affecting the plant's performance under the Consent Decree.

Rhodia Inc. - Houston #8 and #2 Plants Consent Decree Semi-Annual Report for Period Covering January 1 to June 30, 2013 Civil Action No.: 2: 07-CV-134-WCL

4. Status of Permit Applications

3. 4

Houston Title V air permit O-03049 was approved on June 28, 2012. Requirements for compliance with 40 CFR Part 60.83(a)(1) (sulfuric acid mist) Regen #2 and compliance with the Consent Decree SO₂ emission rates for Regen #2 have been included as conditions in the Title V air permit. An amendment to the Houston Title V air permit is being prepared to allow for the start-up of the SO₂ abatement system in Houston #2.

The application to amend air permit 4802 for installation of the SO₂ abatement system in Houston #2 was approved by TCEQ and USEPA on February 10, 2012. The permit amendment to the RCRA permit authorizing installation of the SO₂ abatement system in Houston #2 was approved by TCEQ on October 18, 2012.

Operation and Maintenance Work

The plant does not currently have any operation or maintenance work pending as a result of any of the conditions of the Consent Decree.

5. Reports to Agencies

Rhodia has installed a dual range SO_2 and a new O_2 CEMS for the Houston #8 in 2008. The SO_2 and O_2 CEMS monitor and record the 3-hour arithmetic average SO_2 emission rate in units of lbs. SO_2 per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

A relative accuracy test (RATA) was conducted on February 25, 2013 on the stack SO_2 and O_2 . A cylinder gas audit was conducted on May 8, 2013 on the stack SO_2 and O_2 . The CEMS passed these tests.

The plant submitted Excess Emission Reports for SO₂ per 40 CFR 60.7(c)-(d) and Data Assessment Reports for SO₂ CEMS per 40 CFR Part 60, Appendix F for the first and second calendar quarters of 2013 to the US Environmental Protection Agency (USEPA) and TCEQ.

A copy of the semiannual report is attached which includes the results of the cylinder gas audits.

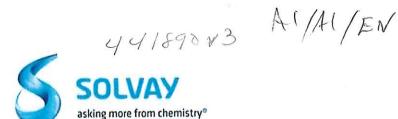
Rhodia Inc. - Houston #8 and #2 Plants Consent Decree Semi-Annual Report for Period Covering January 1 to June 30, 2013 Civil Action No.: 2: 07-CV-134-WCL

I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and their attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

Name/Position: William McConnell/Plant Manager of Baytown and Houston Plants

Signature

Date:



110000460901 GEN-AA TX

Solvay USA Inc. **Houston Plant**

Air/Toxics & Inspection

Coordination Branch

6EN-A

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 1071)

April 4, 2014

Mr. Jeff Robinson Chief. Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Solvay Benzene NESHAP, Subpart FF, Quarterly Report

January 1, 2014 to March 31, 2014 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Solvay receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Solvay submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Solvay USA Inc. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the average temperature of the gas stream in the combustion zone for the TS Vapor Combustor was <50°F below design temperature when being used as the control device for the TS storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Marie

Plant Manager

Air Section Manager, TCEQ, Region 12 cc:

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Solvay USA Inc. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: March 31, 2014

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes Except as Noted	
Quarterly visual inspections of tank covers	x Yes Except as Noted	
Initial and annual Method 21 Hispections of	x Yes Except as Noted	
containers per 61.345(a)(1) Initial and quarterly visual inspections of	x Yes Except as Noted	
containers per 61.345(b) Annual Method 21 inspections of treatment system openings (Regeneration Unit No. 2)	x Yes Except as Noted	
Annual Method 21 inspections of closed vent systems (from tanks to TS vapor combustor and Regeneration Unit No. 2 industrial	x Yes Except as Noted	
Quarterly visual inspections of closed vent systems and control devices (from tanks to TS vapor combustor and Regeneration Unit No. 2 industrial furnace, including the vapor combustor and Regeneration Unit No. 2	x Yes Except as Noted	
industrial furnace) per 61.349(f) Daily inspections of control device continuous monitoring data (temperature of TS vapor combustor and "selected parameter" on Regeneration Unit No. 2 industrial furnace) per 61.354(c)	x Yes Except as Noted	

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

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Air/Toxics & Inspection Coordination Branch 6EN-A

Solvay USA Inc. Houston Plant

CERTIFIED MAIL: RETURN RECEIPT REQUESTED (7011 2000 0001 4575 4195)

February 27, 2014

Mr. Jeff Robinson Air Permits Section Mail Code 6PD-R U.S. EPA – Region VI 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

RE: Benzene Waste Operations NESHAP

Industrial Solid Waste Registration No. 31019 Hazardous Waste Permit No. HW-50095

40 CFR Part 61, Subpart FF EPA ID No. TXD008099079

Dear Mr. Robinson:

Enclosed please find a report for the 2013 calendar year Benzene Waste Operations summary for Solvay USA Inc.'s Houston, Texas facility. Solvay operates a commercial industrial furnace permitted under 40 CFR Part 264 and Part 266 Subpart H by the State of Texas. This report is required under 40 CFR Part 61, Subpart FF-National Emission Standard for Benzene Waste Operations.

We have reviewed the status of each waste stream subject to regulation under this standard. In accordance with section 61.355(a), the Total Annual Benzene (TAB) quantity from this facility's waste operations was 39.7 megagrams for the operating year 2013.

Quarterly fugitive emission monitoring did not identify any emissions >500 ppm as defined in 40 CFR 61.343(a)(1)(i)(A).

Solvay documented all daily visual inspections of the hazardous waste operations area as required in the quarterly inspection requirement as defined in 40 CFR 61.343(c). Visual inspections included sight, smell and sound observations and found no leaks in 2013.

If there are any questions, or if further information is required, please contact me at 713-924-1408.

Sincerely,

W. F. Dickerson

Environmental Manager

Attachment

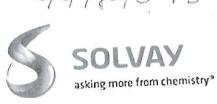
Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012 CC: Air Section Manager, TCEQ, Region 12, Houston
Mr. Bob Allen, Director, Environmental Public Health Division,
Harris County Public Health and Environmental Services
Mr. Arturo Blanco, City of Houston, Bureau of Air Control

GEN-AA

40 CFR 61 Subpart FF - Benzene Annual Report

Solvay USA Inc. Houston-Plant Calendar Year 2013 Annual Benzene Report

$(a)(2) \qquad \qquad \boxed{ \texttt{61.357(a)(3)(i)} \boxed{ \texttt{61.357(a)(3)(ii)} } \boxed{ \texttt{61.357(a)(3)(iii)} } \boxed{ \texttt{61.357(a)(3)(iv)} } \boxed{ \texttt{61.357(a)(3)(v)} } \boxed{ \texttt{61.357(a)(3)(v)} } \boxed{ \texttt{61.357(a)(3)(v)} }$	> = = = = = = = = = = = = = = = = = = =	Y Y 361.8 0-10 10 0.0	10-200	0-10	10,000-50,000	0-10.000	1.300	500-6,000	0	0-1.000	10-2,000 2,000	Y=Yes, N=No Y=Yes, N=No TOTAL
61.357(a)(2)		>	>	>	<u></u>	>	>	>	>-	>-	У	Y=Yes, N=No Y
61.35	Waste Stream	9109003	9104004			0.09020	1301002	1303001	1205001	7 0912006	9405021	
	Lbs. of Waste Burned	797,600	506,790	319,140	1,374,040	448,060	437,160	1,446,282	870,240	4,196,167	303,020	



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Air/Toxics & Inspection

6EN-A

January 29, 2014

Via FedEx

Chief, Environmental Enforcement Division **Environment and Natural Resources** Division U.S. Department of Justice Box 7611 Ben Franklin Station Washington. DC 20044-7611 Jan Gerro U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6RCEA Dallas, TX 75202

Coordination Branch Phillip Brooks U.S. Environmental Protection Agency HQ William Jefferson Clinton Building 1200 Pennsylvania Avenue, N.W. Mailcode 2242A Washington DC 20460

Himanshu Vyas U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6ENAT Dallas, TX 75202

Re: Rhodia Inc. - Consent Decree Semi-Annual Report U.S. v. Rhodia, USDC (N.D. Ind.) Case No. 2: 07-CV-134-WCL

Ladies and Gentlemen:

In accordance with Section VII of the Consent Decree ("CD") entered in the above-entitled matter, enclosed please find the Semi-Annual Report for Rhodia's Houston, Texas facility. The Report satisfies, together with the other supporting documents enclosed, Rhodia's obligation to report on certain matters under the CD within 30 days after the end of each half calendar year (see CD ¶¶ 21-23).

If you have any questions or wish to discuss this submittal, please do not hesitate to contact me.

Thank you.

Sincerely yours,

JSL:vkd Enclosure

Bcc: F. Sardo

Solvay USA Inc. - Houston #8 and #2 Plants

Consent Decree

Semi-Annual Report for Period Covering July 1 to December 31, 2013 Civil Action No.: 2: 07-CV-134-WCL

Effective Dates: 1.

- a. Houston #8 July 1, 2009
- b. Houston #2 April 1, 2014
- Status of Construction or Compliance Measures Necessary to Meet Emissions 2. Limits.

The plant has now completed the construction and implementation of all compliance measures necessary to meet the CD emission limits for #8 Unit. The SO₂ abatement was started up on November 19, 2008.

Construction has commenced for the Houston #2 SO₂ abatement system. The foundation has been poured, structural steel installed, the scrubber vessel has been set, pumping and piping installation has been installed and currently instrumentation is being installed.

- Compliance Issues and Proposed or Implemented Solutions 3.
 - (a) Houston #8 Long-Term SO₂ Limit of 1.70 lbs/ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #8 unit operated below the permitted 1.70 lbs SO₂/ton of acid produced from January 1, 2013 to December 31, 2013.
 - (b) Houston #8 Short-term SO₂ limit of 3.00 lbs/ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.
 - (c) During the reporting period, the plant has not encountered any problems, and does not anticipate encountering any problems, with any of the conditions of the Consent Decree or any applicable permits or any other event affecting the plant's performance under the Consent Decree.

Solvay USA Inc. - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering July 1 to December 31, 2013 Civil Action No.: 2: 07-CV-134-WCL

4. Status of Permit Applications

Houston Title V air permit O-03049 was approved on June 28, 2012. Requirements for compliance with 40 CFR Part 60.83(a)(1) (sulfuric acid mist) Regen #2 and compliance with the Consent Decree SO₂ emission rates for Houston #2 have been included as conditions in the Title V air permit.

The permit amendment to the RCRA permit authorizing installation and start-up of the SO_2 abatement system in Houston #2 was approved by TCEQ on October 18, 2012.

Operation and Maintenance Work

The plant does not currently have any operation or maintenance work pending as a result of any of the conditions of the Consent Decree.

5. Reports to Agencies

Solvay has installed a dual range SO_2 and a new O_2 CEMS for the Houston #8 in 2008. The SO_2 and O_2 CEMS monitor and record the 3-hour arithmetic average SO_2 emission rate in units of lbs. SO_2 per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

Cylinder gas audits on the stack SO2 and O2 CEMS were conducted on September 17, 2013 and October 12, 2013. The CEMS passed these tests.

The plant submitted Excess Emission Reports for SO₂ per 40 CFR 60.7(c)-(d) and Data Assessment Reports for SO₂ CEMS per 40 CFR Part 60, Appendix F for the third and fourth calendar quarters of 2013 to the US Environmental Protection Agency (USEPA) and TCEQ.

A copy of the semiannual report is attached which includes the results of the cylinder gas audits.

Solvay USA Inc. - Houston #8 and #2 Plants Consent Decree Semi-Annual Report for Period Covering July 1 to December 31, 2013

Civil Action No.: 2: 07-CV-134-WCL

I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and their attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

Name/Position: William McConnell/Plant Manager of Baytown and Houston Plants



Solvay USA Inc. Houston Plant

CERTIFIED MAIL; RETURN RECEIPT REQUESTED: (7011 2000 0001 4575 0463)

January 14, 2014

Texas Commission on Environmental Quality Office of Permitting, Remediation and Registration Air Permits Division, MC-163 P.O. Box 13087 Austin, Texas 78711-3087

Subject:

Solvay USA Inc. (CN600125330)

Houston Plant (RN100220581)

Consent Decree (Civil Action No. 2:07CV134 WL)

Air Permit 19282 and PSD-TX-1081

Excess Emission Report for SO₂ per 40 CFR 60.7(c)-(d)

Data Assessment Report for SO₂ and O₂ CEMs per 40 CFR Part 60, Appendix F

Dear Sir or Madam:

In accordance with the Consent Decree referenced above, the Solvay USA Inc. (Solvay) formally Rhodia Inc. Houston No. 8 became subject to 40 CFR Part 60 Subpart H, Standards of Performance for Sulfuric Acid Plants on November 19, 2008. Further, the Consent Decree specifies a SO₂ emission standard that is more stringent than Subpart H and also incorporates an EPA-approved Alternative Monitoring Plan (AMP). As such, the semiannual excess emission report required by 40 CFR 60.7(c)-(d) and the semiannual data assessment report (DAR) required by 40 CFR Part 60 Appendix F, Procedure 1, Section 7 will address compliance with respect to the more stringent CD requirements and the AMP. These reports are attached for the July 1 to December 31, 2013 semiannual reporting period.

The relevant SO₂ standards required by the CD and AMP are as follows:

- Per CD paragraph 11.b.i, emissions of SO₂ are not to exceed a long term limit of 1.70 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per CD paragraph 11.b.ii, emissions of SO₂ are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

As discussed in the AMP, Solvay uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO₂ and % O₂) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b).

Solvay USA Inc.

Page 2

If you have any questions or require additional information, please contact Floyd Dickerson at 713-924-1408.

Sincerely,

millen Jin Toull William McConnell

Plant Manager

Attachment(s)

cc:

Air Section Manager, TCEQ Region 12

Mr. Bob Allen, Director, Harris County Pollution Control

Mr. Arturo Blanco, Bureau Chief of Air Quality Control, Health and Human Services Department, City

Mr. Huimamshu Vyas, EPA Region 6, 1445 Ross Avenue, Suite 1200, Mailcode 6ENAT, Dallas, TX 75202-2733

EPA Region 6, New Source Review Program, 1445 Ross Avenue, Dallas, TX 75202-2733

NSPS Excess Emissions Report July 1 – December 31, 2013

General Information:

General Information:	- 10 B: 11 (00)			
Pollutant:	Sulfur Dioxide (SO ₂)			
Reporting period dates:	July 1 – December 31, 2013			
Emission Limitation:	3.00 lbs/ton short-term, 1.70 lbs/ton long-term			
Address:	8615 Manchester Street, Houston, Texas 77012			
Process Unit Description (Source Unit No):	No. 8 Sulfuric Acid Unit			
Monitor Manufacturer and Model No (Stack SO2):	Ametek Model 920			
Date of Latest CEMS Certification or Audit (Stack):	October 12, 2013			
CEMS span values per the AMP (Stack) (1):	Dual range: Normal: 0 - 500 ppm SO ₂ SSM: 0 - 3,600 ppm SO ₂			

Notes:

⁽¹⁾ Refer to EPA approved Alternative Monitoring Plan for the Houston No. 8 Unit.

NSPS Excess Emissions Report July 1 – December 31, 2013

Emission data summary – Long-Term Limit

ation of excess emissions (as defined per CD and AMP) in reporting period du	
. Startup/shutdown	0 hours
. Control equipment problems	0 hours
. Process problems	0 hours
. Other known causes	0 hours
Unknown causes	0 hours
I duration of excess emission	0 hours
I duration of excess emissions as percent of total source operating time	0%

Emission data summary – Short-Term Limit

Duration of excess emissions (as defined per CD and AMP) in reporting per	NA – limit does not apply durin
a. Startup/shutdown	startup/shutdown
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
Total duration of excess emission	0 hours
Total duration of excess emissions as percent of total source operating time	0%

NSPS Excess Emissions Report July 1 - December 31, 2013

CEMS downtime in reporting period due to:	
	0 hours
Monitor equipment malfunctions	0 hours
. Non-Monitor equipment malfunctions	102.6 hours
. Quality assurance calibration	0 hours
I. Other known causes	0 hours
e. Unknown causes	102.6 hours ⁽¹⁾
. Total CEMS Downtime . Total CEMS Downtime as percent of total source operating time	2.32 %

ack O ₂ Analyzer	
CEMS downtime in reporting period due to:	0 hours
. Monitor equipment malfunctions	0 hours
o. Non-Monitor equipment malfunctions	102.6 hours
c. Quality assurance calibration	0 hours
d. Other known causes	0 hours
e. Unknown causes	102.6 hours (1)
. Total CEMS Downtime	2.32 %
. Total CEMS Downtime as percent of total source operating time	

⁽¹⁾ The Houston #8 Unit followed procedures specified in an EPA approved Alternative Monitoring Plan (AMP) for CEMS malfunctions. In accordance with the AMP, during CEMS malfunctions lasting more than 24 continuous hours Rhodia generally:

Conducted sampling with hand held monitors when the stack SO_2 and O_2 CEMS malfunctioned.

NSPS Excess Emissions Report January 1 – June 30, 2013

Data Assessm Analyzer/	ent Reports (I	DARs) per 40 CFR Ac	curacy Assessme	ent	Any out-of- control periods
Pollutant/Units	Period	Type (RATA, CGA, or RAA)	Any Out-of- Control Periods?	Notes	for Calibration Drift Assessment?
Stack SO ₂ , ppm	3Q13 4Q13	CGA CGA	No No	Report enclosed Report enclosed	No No No
Stack O ₂ , %	3Q13 4Q13	CGA CGA	No No	Report enclosed Report enclosed	No

Describe any changes since last quarter in CEMS, process or controls:

There have been I	no changes in the CEMS,	process, or controls since t	he unit was started on November 19, 20	. 80
÷				

******* Certification Statement for Summary Report per 40 CFR 60.7(d)**********

I certify that the information contained in this report is true, accurate, and complete.

William McConnell	
William McConnell Name of Responsible Official	
Willin J. M. Tonch	
Signature	
Plant Manager Title	
1.4/5.14	



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

Date: 9/17/13 Ti Serial Number: VE-920	me: <u>9:3</u> 0-8700-3	30A	Signa	ician:_fu	& Barri	220
Cylinder ID number Date of Certification	ALM 036 5/20/11	115	ALM00:			
Type of certification (c.g. EPA Protocol 1	EPA PIL		EPA Pro	tocol		
or CRM).	Trial 1		Trial 2		Trial 3	
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value	5,09	15,3	5.09	15.3	5.09	15.3
Ca (ppm) CEM Response	4.98	15.5	5,01	15.5	5.00	15.3
value C_m (ppm) Accuracy A (% or ppm)	-2.161%	1.307%	-1.5727	1.307%	-1.768%	1.307%

where $A = (C_m - C_a) \times 100$ C_a



Air Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

· Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006 .

RHODIA INC LAB

11426 FAIRMONT PKWY LA PORTE, TX 77571

8615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115

Certification Date:

20May2011

20May2013 Exp. Date:

Cylinder Number: Cylinder Pressure***

1934 PSIG

LAP0041227 Batch No:

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY**

TRACEABILITY

SULFUR DIOXIDE *

PPM

+7-1%

Direct NIST and VSL

OXYGEN NITROGEN BALANCE

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997,

REFERENCE STANDARD

TYPE/SRM NO.

EXPIRATION DATE 15Ján2012

CYLINDER NUMBER

CONCENTRATION 255.5 PPM

COMPONENT SULFUR DIOXIDE OXYGEN

NTRM 0260 NTRM 2350

:01May2013

KAL003774 K026427

23.50 %

ANALYTICAL PRINCIPLE

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

ETIR/IMG-09-149 SERVOMEX/MODEL 244A/701/716 DATE LAST CALIBRATED 12May2011

Response Unit: PPM

T1 = 274.2461

25Apr2011

PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

R1 = 0.94000

Z2 = 0.00000

T3=0.61140

Second Triad Analysis

Calibration Cuive

SULFUR DIOXIDE *

Response Unit; PPM

Date: 13May2011 T1 = 276.1649R1=256.6585 Z1 = 0.06656T2 = 275.4641Z2=0.11493 R2=256.8202 R3=258.8364 T3=275.6497

Z3=0.15836 Avg. Concentration:

Date: 19May2011

Avg. Concentration:

Z1=0,00000

R2=0.94000

Z3 = 0.00000

OXYGEN

274.1 PPM

Response Unit: VOLTS

15.27

T1=0.61100

72 = 0.61140

R3=0,94000

Date: 20May2011 21=-0.32212

R1 = 256.5447 Z2=0.15853 R2=255,6349 73 = 0.33653Avg. Concentration:

T2=274.6593 R3=256.5102 T3=274.7581

Concentration = A + Bx + Cx2 + Dx3 + Ex4 r=9.99998E-1 A=0,00000E+0

Constants:

C=3,00000E-6 B=9.99951E-1 E = 0.00000E + 0D=0.00000E +0

Concentration = A + Bx + Cx2 + Dx3 + Ex4 r = 9999987

Constants:

A = .000249421

B=24,9768807

Ċ≒

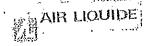
Special Notest

660 CGÁ DÉW POINT 40 F

APPROVED BY

Ramien JR

Page __1_0f-1



Air Liquide America Specially Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77671

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free | Wulti-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-005

11426 FAIRMONT PKWY LA PORTE, TX 77571

B615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997,

ALM002590 Cylinder Number: 1936 PSIG

Certification Date:

20May2011

20May 2013 Exp. Date:

LAP0041255 Batch No:

Cylinder Pressure***

CERTIFIED CONCENTRATION (Moles)

PPM

ACCURACY**

TRACEABILITY

COMPONENT SULFUR DIOXIDE *

1-1-1% +1-1%

Direct NIST and VSL

OXYGEN NITROGEN

BALANCE

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD

TYPE/SRM NO. NTRM 0260

EXPIRATION DATE

15Jan2012 01May2013 CYLINDER NUMBER KAL003774 K028427

CONCENTRATION 255.5 PPM 23,50

COMPONENT SULFUR DIOXIDE

OXYGEN

NTRM 2350 INSTRUMENTATION INSTRUMENT/MODEL/SERIAL/

FTIR//MG-09-149 SERVOMEX/MODEL 244A/701/716 DATE LAST CALIBRATED 12May2011

25Apr2011

ANALYTICAL PRINCIPLE

FTIR PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Curve

SULFUR DIOXIDE *

Response Unit: PPM Date: 13May2011

T1=123.9645 R1=253.7089 21=0.01023 T2 = 124.0038Z2=0.07108 B2 = 253.7350

Avg. Concentration:

R3=253,8609 T3=124.1083

23=0.09222 124.8

OXYGEN

Résponse Unit: VOLTS Date: 19May2011 T1=0,20370 R1=0.94000 Z1 = 0.00000

T2=0.20400 22=0.00000 R2=0.94000-R3=0.94000 T3=0.20400 Z3 = 0.00000

Avg. Concentration:

5.093

Response Unit: PPM Date: 20May2011

T1=123.9691 R1 = 253.7032Z1 = 0.00195T2 = 124.0436Z2=0.10441 R2=253,8925 B3=254.0720 T3=124.0462 23 = 0.12747

PPM Avg. Concentrations

Concentration = A+Bx+Cx2+Dx3+Ex4 1=9.99985E-1

0+300000E+0 Constants: C=0,00000E+0 B=9.94461E-1

E = 0.000000E + 0D=0.00000E+0

Concentration = A+Bx+Cx2+Dx3+Ex4

r = .9999987

Constants:

A = .000249421C=

B=24.9768607

Special Notes:

660 CGA DEW POINT 40 F

APPROVED BY

Ramien JR



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

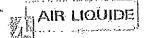
(8)

Date: 9/17/13 Time: 9:30
Serial Number: VE-920-8700-2

Technician: Prody Barraza
Signature: P. B.

ALM 03 5/20/1		ALM OO	2590		
		ALM,002590			
	5/20/11		5/20/11		
EPA Protocol		EPA Protocol			
1		•			
Trial I		Tria	al 2	Trial 3	
Audit	Audit	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
125	274	125	274	125	27.4
126	277	125	276	124	277
\ <u></u>	1.095%	090	.730%	800%	1.095%
	Audit Point 1 125 126	Trial I Audit Audit Point 2 125 274 126 277	Trial 1 Trial Audit Audit Audit Point 1 Point 2 Point 1 125 274 125 126 277 125	Trial 1 Trial 2 Audit Audit Audit Point 1 Point 2 125 274 125 274 126 277 125 276	Trial 1 Trial 2 Trial Audit Audit Audit Audit Point 1 Point 2 Point 1 Point 2 Point 1 125 274 125 274 125 126 277 125 276 124

where $A = (C_m - C_a) \times 100$ C_a



Alr Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

<u>Customer</u> RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document #: 41649621-005 11426 FAIRMONT PKWY LA PORTE, TX 77671

8615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALW002590 Cylinder Number: 1936 PSIG Cylinder Pressure***:

Certification Date:

20May2011

Exp. Date: Batch No:

20May2013 LAP0041255

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY** 4/-1%

TRACEABILITY

SULFUR DIOXIDE *

Direct NIST and VSL

OXYGEN-NITROGEN

BALANCE

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD

TYPE/SRM NO. NTRM 0260

NTRM 2350

EXPIRATION DATE 16Jan2012 01May2013

CYLINDER NUMBER KAL003774

K026427

CONCENTRATION

255.5 PPM 23,50

DATE LAST CALIBRATED

COMPONENT SULFUR DIOXIDE

OXYGEN

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL# FTIR//MG-09-149 SERVOMEX/MODEL:244A/701/716

12May2011 25Apr2011

ANALYTICAL PRINCIPLE FTIR

PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Curve

SULFUR DIOXIDE *

Response Unit: PPM Date: 13May2011

R1 = 253.7089 T1 = 123.964571 = 0.01023T2=124,0038 Z2 = 0.07108R2=253.7360 T3 = 124.1083

23=0.09222 Avg. Concentration:

R3=263.8609 PPM 124.8

Date: 20May2011 Z1 = 0.00195

R2=253,8925

Responsé Unit: PPM R1 = 253,7032

11 = 123,9691T2=124.0436

22 = 0.10441R3 = 254.0720T3=124.0462

23=0.12747 PPM Avg. Concentration:

Concentration = A + Bx + Cx2 + Dx3 + Ex4

r=9,99985E-1 Constants:

A=0.00000E+0

B = 9.94451E-1

C=0.00000E+0 E=0.00000E+0 D = 0.00000E + 0

OXYGEN

Response Unit: VOLTS Date: 19May2011

R1 = 0.94000Z1=0.00000 R2=0,94000 22 = 0.00000

T1=0.20370 T2 = 0.20400R3=0.94000 T3 = 0.20400

Z3=0.00000 Avg. Concentration:

5.093

Concentration = A + Bx + Cx2 + Dx3 + Ex4

r = .9999987Constants:

A = .000249421C=

B=24,9768607

Special Notes:

660 CGA DEW POINT 40 F

APPROVED BY:

Ramien JR Page 1-01-1



Air Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

11426 FAIRMONT PKWY

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006 -

8616 MANCHESTER HOUSTON TX 77012

LA PORTE, TX 77571

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115

Certification Date:

20May2011

20May2013 Exp. Date: Batch No:

LAPO041227

Cylinder Number: Cylinder Pressure***

1934 PSIG

ACCURACY**

TRACEABILITY

COMPONENT

CERTIFIED CONCENTRATION (Moles) PPM

+/-1% -1-1-1-% Direct NIST and VSL

SULFUR DIOXIDE * OXYGEN

BALANCE

NITROGEN

*** Do not use when cylinder pressure is below 150 pslg. ** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997,

REFERENCE STANDARD

TYPE/SRM NO.

EXPIRATION DATE 15Jan2012

CYLINDER NUMBER KAL003774 K026427 01May2013

CONCENTRATION

255.5 PPM 23.50 %

COMPONENT SULFUR DIOXIDE OXYGEN

NTRM 0260 NTRM 2350

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

FTIR//MG-09-149

SERVOMEX/MODEL 244A/701/716

DATE LAST CALIBRATED

12May2011 25Apr 2011

ANALYTICAL PRINCIPLE

FTIR PARAMAGNETIC

ANALYZER READINGS

[Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient]

First Triad Analysis

Second Triad Analysis

Calibration Cuive

Concentration = A + 8x + Cx2 + Dx3 + Ex4

SULFUR DIOXIDE *

Response Unit: PPM Date: 13May2011 T1 = 276.1549

H1=256,6585 Z1 = 0.06656T2 = 275.464122 = 0.11493R2=256.8202

R3=258.8364 T3=275.6497 23=0.15836 PPM 274.1

Avg. Concentration:

OXYGEN

Response Unit: VOLTS Date: 19May2011 T1=0.61100 R1 = 0.94000 z1 = 0.000000T2≓0.61140 22=0.00000

R2=0.94000 R3 = 0.94000T3=0.61140 Z3=0.00000

15.27 Avg. Concentration:

Response Unit: PPM Date: 20May2011 T1 = 274,2461R1=256.5447

21=-0,32212 T2=274.6593 Z2 = 0.15853R2=255,6349

R3 = 256.5102 T3 = 274.768123 = 0.33653-PPM Avg. Concentration:

B=9.99951E-1 D=0.00000E+0

Concentration=A+Bx+Cx2+Dx3+Ex4

r= 9999987

r=9.99998E-1

Constants:

Constants:

A = .000249421

A=0,00000E+0

E = 0.00000E + 0

C=3.00000E-6

B=24.9768807 Ç:a

Special Notes:

680 CGÁ ĎEW POINT 40 F

APPROVED BY

Ramien JR

Page__1-of-



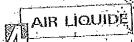
Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):	2	(8)
Date: 9/17/13 Time:/0;20A Serial Number:		Technician: Rody Barraza. Signature: R. B.

				•		
Cylinder ID number	ALM 045898		ALM 058443			
Date of Certification	7/18/2011		7/18/2011			
Type of certification	EPA Protocol		EPA Protocol			
(e.g. EPA Protocol 1			1			
or CRM).	Trial 1		Trial 2		Trial 3	
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	909	1970	909	1970	909	1970
CEM Response value C_m (ppm)	900	1965	906	1971	908	1972
Accuracy A (% or ppm)	990%	-,254%	-,330%	.051%	-,110%	.102%

where $A = (C_m - C_n) \times 100$ C_a



Air Liquide America Specially Gases LLC



COMPLIANCE CLASS.

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No. 4501452424

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document, 423031,15-003

Customer RHODIA INC. STOREROOM

ATTN PAUL BARNETT 8615 MANCHESTER

ANALYTICAL INFORMATION Gas Type: SO2

This certification was performed according to EPA Traceability Protocolifor Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALMO45898

Cettification Date:

1,8Jul2011

17Jul2014

Cylinder Number: Cylinder Pressure***

1970 PSIG

LAP0044990 Batch No.

CERTIFIED CONCENTRATION (Moles)

ACCURACY**

TRACEABILITY

COMPONENT SULFUR DIOXIDE * NITROGEN

NIST and VSL

Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol procedures , September 1997,

REFERENCE STANDARD

TYPE/SRM NO.

EXPIRATION DATE 01 ปีข้หั2016

CYLINDER NUMBER

COMPONENT

NTRM 1662

975,0

SULFUR DIOXIDE

INSTRUMENTATION. NSTRUMENT/MODE

FTIR/IMG-09-149

LAST ON BERATED 08.Jul2011

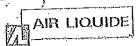
ANALYTICAL PHINCIPLE

Special Notes:

POINT TO F. CGA 660 RDIAORTY005

APPROVED BY

,1°of.1 Page



Air Liquide America Specially Gases LLC



COMPLIANCE CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID; A32011 .

P.O. No.: 4501452424

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document #1 42803115-004

.11426 FAIRMONT PKWY LA PORTE, TX 77571

RHODIA INC. STOREROOM

ATTN PAUL BARNETT 8615 MANCHESTER HOUSTON TX 77012

*ANALYTICAL INFORMATION Gas Type : SO2

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

Cylinder Number:

Cylinder Pressure***:

1963 PSIG

19Jul2011

Exp. Date:

18Jul2014

Cylinder Number: Cylinder Pressure***

Batch No.

LAP0045081

CERTIFIED CONCENTRATION (Moles)

TRACEABILITY NIST and VSL

COMPONENT SULFUR DIOXIDE * NUTROGEN

1,970

ACCURACY***

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol procedures , September 1997.

REFERENCE STANDARD

TYPE/SRM NO. NTRM 1664

EXPIRATION DATE 020ct2011

CYLINDER NUMBER ALM043439

CONCENTRATION

COMPONENT SULFUR DIOXIDE

INSTRUMENTATION VINSTRUMENT/MODEL/SERIAL/#: FTIR//MG-09-149

, DEW POINT 40 F. GGA GGO RDIAORTYCOG



Eco Services - Houston

02

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

(8)

Date: 10/12/13 Time: 1591M Serial Number: VE-930-8700-2 Technician: Paul Barnett Signature: Paul Barnett

	,					
Cylinder ID number	ALM BE	72590	ALM 036115			
Date of Certification	5-20-2011		5-20-2011		·	
Type of certification	EPA-Protocal I 125 ppm SO2 504 9602		EPA-Protocol I			
(e.g. EPA Protocol 1 or CRM).	125 ppm SO2 5-09		274ppm Suz			
	Trial 1		Tria	d 2	Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2_	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	5.09	15-3	5.09	15.3	5.09	15,3
CEM Response value C_m (ppm)	5.00	15.4	5-082	15.4	5-02	15-4
Accuracy A (% or ppm)	09%	+.1%	-,07%	+.1%	07%	+.1%

where $A = (C_m - C_a) X 100$ C_a



Air Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006 -

RHODIA INC LAB

11426 FAIRMONT PKWY LA PORTE, TX 77671

8615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115

Certification Date:

20May2011

Exp. Date:

20May2013 LAP0041227

Cylinder Number: Cylinder Pressure***

1934 PSIG

Batch No:

COMPONENT

CERTIFIED CONCENTRATION (Moles) PPM

ACCURACY** +/- 1% +/=1%

TRACEABILITY Direct NIST and VSL

SULFUR DIOXIDE *
OXYGEN

NITROGEN

BALANCE

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the redultements of EPA Protocol Procedure G1, September 1997,

REFERENCE STANDARD

TYPE/SRM NO.

EXPIRATION DATE 15Jan2012

CYLINDER NUMBER KAL003774

CONCENTRATION 255.5 PPM

COMPONENT SULFUR DIOXIDE

NTRM 0260 :01May2013 NTRM 2350

K026427

23.50 %

OXYGEN

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

ETIR//MG-09-149 SERVOMEX/MODEL 244A/701/716 DATE LAST CALIBRATED 12May2011 25Api2011

ANALYTICAL PRINCIPLE PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Cuive

SULFUR DIOXIDE *

Response Unit; PPM Date: 13May2011 T1 = 276.1549 R1=256:6585 Z1 = 0.06656Z2 = 0.11493R2 = 256.8202

Z3=0,15836 Avg. Concentration:

T3 = 275.6497 274.1

T2 = 275.4641

R3 = 258,8364 PPM

Z3=0,33653 Avg. Concentration:

Date: 20May2011

21=-0.32212

R2=255,6349

T1 = 274.2461 R1=255,5447 T2=274,6593 . 22=0.15853 R3=256.5102 T3=274.7681

Response Unit: PPM

Concentration = A + Bx + Cx2 + Dx3 + Ex4

r=9,99998E-1

Constants: B≑9,99951E-1 A=0,00000E+0

D=0.00000E+0

C=3,00000E-6. E=0.00000E+0

OXYGEN

Date: 19May2011 $Z_1 = 0.00000$

Avg. Concentration:

R2=0,94000

Z3=0.00000

Response Unit: VOLTS Ŗ1 ≈ 0.94000 Z2 = 0.00000

T3=0.61140 15.27

T1=0.61100 T2=0.61140 R3=0,94000

Concentration=A+Bx+Cx2+Dx3+Ex4

r= 9999987 Constants:

A = .000249421 Ċ≔

B=24,9768807

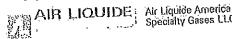
Special Notes:

660 CGA DEW POINT 40 F

APPROVED BY

Ramien JR

Page___1-0f-1



Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

si kang pang tan katikan kang kalan kananda kanan kanan mangkan basi kanan kanan sa sa sa sa sa sa sa sa sa sa

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-005

8615 MANCHESTER

11426 FAIRMONT PKWY LA PORTE, TX 77571

HOUSTON TX 77012 US

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM002590

Certification Date:

20May2011

20May 2013 Exp. Date:

Cylinder Number: Cylinder Pressure***

1936 PSIG

LAPO041255 Batch No:

COMPONENT SULFUR DIOXIDE * CERTIFIED CONCENTRATION (Moles)

ACCURACY **

TRACEABILITY

PPM

+/-1%

Direct NIST and VSL

OXYGEN NITROGEN BALANCE

*** Do not use when cylinder pressure is below 150 psig.

* Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD

TYPE/SRM NO. NTRM 0260

EXPIRATION DATE

15Jan2012 01May2013. CYLINDER NUMBER

KAL003774 K026427

CONCENTRATION

255.5 PPM % 23,50

COMPONENT SULFUR DIOXIDE OXYGEN

NTRM 2350 INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

FTIR//MG-09-149 SERVOMEX/MODEL 244A/701/216 DATE LAST CALIBRATED

Response Unit: PPM

12May 2011 28Apr2011

ANALYTICAL PRINCIPLE

PARAMAGNETIC

ANALYZER READINGS

[Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Curve

SULFUR DIOXIDE *

Response Unit: PPM Date: 13May2011 T1 = 123.9645R1 = 253.708921 = 0.01023T2 = 124.003822=0.07108 R2=253.7350

Z3=0.09222

T3 = 124.1083

R3 = 253.8609PPM

Avg. Concentration:

124.8

Date: 20May2011 21=0.00195 B2=253,8925 23=0.12747 Avg. Concentration:

R1 = 253.7032Z2 = 0.10441T3=124,0462

T1 = 123,9891T2 = 124.0436R3=254.0720

PPM

Concentration = A + Bx + Cx2 + Dx3 + Ex4 r=9.99985E-1 A=0.00000E+0 Constants:

B=9.94451E-1 D = 0.000000E + 0

C=0.00000E f0 E=0,00000E+0

OXYGEN

Date: 19May2011 Z1 = 0.000000

Response Unit: VOLTS R1 = 0.9400022 = 0.00000

n2=0.94000 Z3⇔0.00000 T3 = 0.20400

T1 = 0.20370T2 = 0.20400R3=0.94000

Avg. Concentration:

5.093

Concentration = A+Bx+Cx2+Dx3+Ex4

r = .9999987Constants:

A = 000249421

B=24.9768607 .C == E=

Special Notes:

660 CGA DEW POINT 40 F

APPROVED BY Ramlen JR

Page-



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

(8)

Date: 10/12/13 Time: 139 pm Serial Number: VE - 920 - 8700 - 2 Technician: Paul Burnett Signature: Paul Burnett

Cylinder ID number	ALM \$36115		ALM DO 2590			
Date of Certification	5-20-2011		5-20-2011			
Type of certification	EPA Protocol I		EPA Protocol 1			
(e.g. EPA Protocol 1						
or CRM).	272 ppm 502		125 ppm SO2			
, , , , , , , , , , , , , , , , , , ,	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	125	12/14	500/25	5 274	125	274
CEM Response value C_m (ppm)	124	272	125	271	125	272
Accuracy A (% or ppm)	-1 ppm	-2 ppm	Oppn	-3ppm	Оррт	-2ррт

where $A = (C_m - C_a) \times 100$ High Range C_a



IDE: Air Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77671

· Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006 .

11426 FAIRMONT PKWY LA PORTE, TX 77571

8615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115

Certification Date:

20May2011 -

20May2013 LAP0041227

Cylinder Number: Cylinder Pressure***

1934 PSIG

Batch No:

TRACEABILITY

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY** +1-1%

Direct NIST and VSL

SULFUR DIOXIDE *

#*|*=1%

OXYGEN NITROGEN

BALANCE

* * * Do not use when cylinder pressure is below 160 pslg.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997,

REFERENCE STANDARD

TYPE/SRM NO. NTRM 0260

EXPINATION DATE 15Jan2012

:01May2013

CYLINDER NUMBER KAL003774 K026427

CONCENTRATION 255.5 PPM 23.50 %

COMPONENT SULFUR DIOXIDE OXYGEN

NTRM 2350 INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

FTIR//MG-09-149 SERVOMEX/MODEL 244A/7017716 DATE LAST CALIBRATED

12May2011 26Apr 2011

ANALYTICAL PRINCIPLE

PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Cuive

SULFUR DIOXIDE *

Response Unit: PPM Date: 13May2011 T1=276.1549 B1 = 256,6585 Z1 = 0.06666T2 = 276.4641

Z2=0.11493 R2=256,8202 T3 = 276.6497 23=0.15836

Avg. Concentration:

R3 = 258.8364 PPM

R2=255,6349 23=0.33653 Avg. Concentration:

Date: 20May2011

21=-0.32212

T1 = 274.246TR1 = 255,544772=0.15853 T3=274.7581

Response Unit: PPM

T2=274,5593. R3 = 256,5102

Concentration = A + Bx + Cx2 + Dx3 + Ex4 r=9.99998E-1

Constants:

A=0.00000E+0 C=3'00000E-8 B=9.99951E-1 E=0.00000E+0 D=0.00000E+0

Concentration=A+Bx+Cx2+Dx3+Ex4 OXYGEN r=.9999987 Response Unit: VOLTS A = .000249421 Constants:

C=

B=24,9768807

Z1 = 0.000000R2=0.94000 Z3 = 0.000000

Date: 19May2011

B1 = 0.94000Z2 = 0.00000T3=0.61140

T1 = 0.61100T2=0.61140 R3=0.94000

Avg. Concentration:

Special Notest

660 CGA DEW POINT 40 F

APPROVED B

Ramien JR



Air Liquide America Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-005 11426 FAIRMONT PKWY

8615 MANCHESTER

LA PORTE, TX 77671

HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM002590

Certification Date:

20May2011

Exp. Date: Bătch No:

20May2013 LAP0041255

Cylinder Number: Cylinder Pressure***:

1936 PSIG

COMPONENT SULFUR DIOXIDE * CERTIFIED CONCENTRATION (Moles)

ACCURACY**

TRACEABILITY Direct NIST and VSL

OXYGEN-

NITROGEN

BALANCE

** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD

TYPE/SRM NO. NTRM 0260

NTRM 2350

EXPIRATION DATE

15Jan2012 01May2013 CYLINDER NUMBER

KAL003774 K028427

CONCENTRATION 255.5 PPM

23.50

COMPONENT SULFUR DIOXIDE

OXYGEN

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL# FTIR//MG-09-149 SERVOMEX/MODEL 244A/701/716

DATE LAST CALIBRATED

12May2011 25Apr2011

ANALYTICAL PRINCIPLE

FTIR PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Calibration Curve

SULFUR DIOXIDE *

Responsé Unit: PPM Date: 13May2011

T1=123.9645 R1 = 253.7089

Z1 =0.01023 62 = 253.7350Z3=0.09222

OXYGEN :

Z1 = 0.00000

R2=0,94000

Z3 = 0.00000.

Z2≈0.07108 T3=124.1083

R1 = 0.94000

72=0.00000

T3≃0,20400

Avg. Concentration:

Date: 19May2011

Avg. Concentration:

124.8

Response Unit: VOLTS

5,093

12=124,0038

T1 = 0.20370

T2 = 0.20400

R3=0,94000

H3=263.8609

PPM

Z1 = 0.00195R2=253,8925 Z3=0.12747

Date: 20May2011

R1 = 253.7032Z2=0.10441 T3=124.0462

T2=124.0436 R3 = 254.0720

Responsé Unit: PPM

T1=123,9691

124.8 Avg. Concentration:

Concentration = A + Bx + Cx2 + Dx3 + Ex4

r=9,99985E-1

Constants: B = 9.94451E-1D = 0.00000E + 0. $A = 0.00000E \pm 0$ C=0.00000E+0 E=0.00000E+0

Concentration = A + Bx+ Cx2 + Dx3 + Ex4 r=.9999987

A = .000249421Constants: C⇔

8=24,9768607

Special Notes:

660 CGA_DEW POINT 40 F

APPROVED BY:

Ramlen JR



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

(8)

Date: 10/12/13 Time: 305 pm Serial Number: VE - 920 - 8100 - 2 Technician: <u>faul Barnett</u> Signature: <u>Aul Barnett</u>

Cylinder ID number	ALM 64	898	ALMOS	8443	[
Date of Certification	7/18/	20/4	7/19.	12011		
Type of certification	EAA Prote	ocol 1	EPA Pro	tocol 1		
(e.g. EPA Protocol 1						
or CRM).	909	502 pm	1970	APM SUZ		
	Tr	ial 1	Tr	ial 2	Tri	al 3
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value						
C_a (ppm)	909	1970	909	1970	909	1970
CEM Response	,					
value C_m (ppm)	908	1968	909	1970	908	1969
Accuracy A (% or	-1.4.4	2	Quen	0100	1 22.00	1
ppm)	- 1 ppm	- 2 ppin	Offen	Oppm	-1 ppm	-/ ppm

where
$$A = (C_m - C_a) \times 100$$
 Low range



Air Liquide America -Specialty Gases LLC



COMPLIANCE CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fex: 281-474-8419

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501452424

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 42303115-004

11426 FAIRMONT PKWY LA PORTE, TX 77571

Customer RHODIA INC. STOREROOM

ATTN PAUL BARNETT 8615 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: \$02

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM058443

Certification Date:

19Jul2011

Exp. Dato:

18Jul2014

Cylinder Number: Cylinder Pressure***: 1963 PSIG

Batch No:

LAP0045081

COMPONENT SULFUR DIOXIDE * NITROGEN

CERTIFIED CONCENTRATION (Moles)

TRACEABILITY NIST and VSL

PPM BALANCE

Do not use when cylinder pressure is below 150 psig.

* Analytical accuracy is based on the requirements of EPA Protocol procedures , September 1997.

REFERENCE STANDARD
TYPE/SRM NO. EX

NTRM 1664

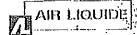
EXPIRATION DATE 020dt2011

CONCENTRATION 2402. PPM

COMPONENT SULFUR DIOXIDE

, INSTRUMENTATION INSTRUMENTIMODELISERIALI: FTIR//MG:09-149

DEVI POINT 40 F. CGA 660 RDIAGRTY008



Air Liquide America Specialty Gases LLC



COMPLIANCE CLASS

Dual-Analyzed Calibration Standard

- 11428 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501452424.

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # , 42303115-003

11426 FAIRMONT PKWY LA PORTE, TX 77571

Customer RHODIA INC. STOREROOM

ATTN PAUL BARNETT 8615 MANCHESTER **JOUSTON TX 77012**

ANALYTICAL INFORMATION Gas Type : SO2

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; Séptember, 1997, Cylinder Number: ALM045898

18Jul2011

Exp. Date:

Cylinder Number: Cylinder Pressure***:

1970 PSIG

Certification Date:

Batch No:

LAP0044990

COMPONENT SULFUR DIOXIDE * NITROGEN

CERTIFIEÓ CONCENTRATION (Moles)

BALANCE

ACCURACY**

TRACEABILITÝ NIST and VSL

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol procedures , Saptember 1997.

REFERENCE STANDARD.

TYPE/SRM NO. EXP

NTRM 1662

EXPIRATION DATE

01Jun2016

CYLINDER NUMBER_. KAL003078

CONCENTRATION 975.0 PPM

INSTRUMENTATION

FILR//MG-09-149

08Ĵul2011

Special Notes:

LDEN POINT 40 F. CGA 660 RDIADRIY005

GARY WRIGHT

SOLVAY
asking more from chemistry®

GEN-AA

RECEIVE

JUL 14.2014

Air/Toxics & Inspection

Coordination Branc 6EN-A

Solvay USA Inc. Houston Plant

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 1385)

July 8, 2014

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Solvay Benzene NESHAP, Subpart FR, Quarterly Report

April 1, 2014 to June 30, 2014 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Solvay receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Solvay submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Solvay USA Inc. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012 Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

William & Mo Cornell

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Solvay USA Inc. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: June 30, 2014

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	ļ
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2) per 61.348(e)(3)(ii)	Except as Noted	
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial	Enterpt as Troted	
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on	1	
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.



441890 VD AMPAY EN T901 RECEIVE W4 JUL 3-1-14

(10000 460 Gal

Air Toxics & Inspection Coordination Branc'

6EN-A

July 30, 2014 Via FedEx

Chief, Environmental Enforcement Division **Environment and Natural Resources** Division U.S. Department of Justice Box 7611 Ben Franklin Station Washington. DC 20044-7611 Jan Gerro U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6RCEA Dallas, TX 75202

Phillip Brooks U.S. Environmental Protection Agency HQ William Jefferson Clinton Building 1200 Pennsylvania Avenue, N.W. Mailcode 2242A Washington DC 20460

Himanshu Vyas U.S. Environmental Protection Agency Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6ENAT Dallas, TX 75202

Re: Rhodia Inc. - Consent Decree Semi-Annual Report

U.S. v. Rhodia, USDC (N.D. Ind.) Case No. 2: 07-CV-134-WCL

Ladies and Gentlemen:

In accordance with Section VII of the Consent Decree ("CD") entered in the above-entitled matter, enclosed please find the Semi-Annual Report for Rhodia's Houston, Texas facility. The Report satisfies, together with the other supporting documents enclosed, Rhodia's obligation to report on certain matters under the CD within 30 days after the end of each half calendar year (see CD ¶¶ 21-23).

If you have any questions or wish to discuss this submittal, please do not hesitate to contact me.

Thank you.

Sincerely yours,

Jeffrey S Lang

JSL:vkd Enclosure

Bcc: F. Sardo

Solvay USA Inc. - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering January 1 to June 30, 2014 Civil Action No.: 2: 07-CV-134-WCL

1. Effective Dates:

- a. Houston #8 July 1, 2009
- b. Houston #2 April 1, 2014
- 2. Status of Construction or Compliance Measures Necessary to Meet Emissions Limits.

The plant has now completed the construction and implementation of all compliance measures necessary to meet the CD emission limits for #8 Unit. The SO₂ abatement unit was started up on November 19, 2008.

Construction has been completed and implementation of all compliance measures necessary to meet the CD emission limits for #2 Unit. The SO₂ abatement unit was started up on February 7, 2014.

The plant has completed the implementation of all compliance measures necessary to meet the CD emission limits.

- 3. Compliance Issues and Proposed or Implemented Solutions
 - (a) Houston #8 Long-Term SO₂ Limit of 1.70 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #8 unit operated below the permitted 1.70 lbs. SO₂/ton of acid produced from July 1, 2013 to June 30, 2014.
 - (b) Houston #8 Short-term SO₂ limit of 3.00 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.
 - (c) Houston #2 Long-Term SO₂ Limit of 1.80 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. The start of the Long-Term Limit compliance period is April 1, 2014.
 - (d) Houston #2 Short-term SO₂ limit of 3.00 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. On June 19, 2014, Houston #2 exceeded the 3.00 lbs./ton limit for 2 hours and 35 minutes.

Solvay USA Inc. - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering January 1 to June 30, 2014 Civil Action No.: 2: 07-CV-134-WCL

(e) During the reporting period, the plant has not encountered any problems, and does not anticipate encountering any problems, with any of the conditions of the Consent Decree or any applicable permits or any other event affecting the plant's performance under the Consent Decree.

4. Status of Permit Applications

Houston Title V air permit O-03049 was approved on June 28, 2012. Requirements for compliance with 40 CFR Part 60.83(a)(1) (sulfuric acid mist) Houston #2 and compliance with the Consent Decree SO₂ emission rates for Houston #2 have been included as conditions in the Title V air permit.

Operation and Maintenance Work

The plant does not currently have any operation or maintenance work pending as a result of any of the conditions of the Consent Decree.

5. Reports to Agencies

Solvay has installed a dual range SO_2 and a new O_2 CEMS for the Houston #8 in 2008. The SO_2 and O_2 CEMS monitor and record the 3-hour arithmetic average SO_2 emission rate in units of lbs. SO_2 per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

A relative accuracy test was conducted on the Houston #8 stack SO_2 and O_2 CEMS on March 12, 2014. A cylinder gas audit was conducted on the stack SO_2 and O_2 CEMS were conducted on June 28, 2014. The CEMS passed these tests.

Solvay has installed a dual range stack SO₂ CEMS and a new main gas blower SO₂ CEMS for the Houston #2 in 2014. The SO₂ CEMS monitor and record the 3-hour arithmetic average SO₂ emission rate in units of lbs. SO₂ per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

A relative accuracy test was conducted on the Houston #2 stack SO_2 CEMS on April 7, 2014. The Houston #2 main gas blower CEMS quarterly cylinder gas audits were conducted on April 9, 2014. The CEMS passed the gas audit tests.

Solvay USA Inc. - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering January 1 to June 30, 2014 Civil Action No.: 2: 07-CV-134-WCL

The plant submitted Excess Emission Reports for SO₂ per 40 CFR 60.7(c)-(d) and Data Assessment Reports for SO₂ CEMS per 40 CFR Part 60, Appendix F for the first and second calendar quarters of 2014 to the US Environmental Protection Agency (USEPA) and TCEQ.

A copy of the semiannual report is attached which includes the results of the RATA and cylinder gas audits.

I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and their attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

Name/Position: William McConnell/Plant Manager of Baytown and Housto	n Pla	lan
--	-------	-----

Signature: Mullin Mi-Cornel

Date: 7/17/14



Solvay USA Inc. Houston Plant

CERTIFIED MAIL; RETURN RECEIPT REQUESTED: (7011 2000 0001 4575 1415)

July 14, 2014

Texas Commission on Environmental Quality
Office of Permitting, Remediation and Registration
Air Permits Division, MC-163
P.O. Box 13087
Austin, Texas 78711-3087

Subject:

Solvay USA Inc. (CN604514315) Houston Plant (RN100220581)

Consent Decree (Civil Action No. 2:07CV134 WL)

Air Permit 19282 and PSD-TX-1081 Air Permit 4802 and PSD-TX-1260

Excess Emission Report for SO₂ per 40 CFR 60.7(c)-(d)

Data Assessment Report for SO₂ and O₂ CEMs per 40 CFR Part 60, Appendix F

Dear Sir or Madam:

In accordance with the Consent Decree referenced above, the Solvay USA Inc. (Solvay) formally Rhodia Inc. Houston No. 8 became subject to 40 CFR Part 60 Subpart H, Standards of Performance for Sulfuric Acid Plants on November 19, 2008 and Houston Regen 2 became subject on April 1, 2014. Further, the Consent Decree specifies a SO₂ emission standard that is more stringent than Subpart H and also incorporates an EPA-approved Alternative Monitoring Plan (AMP). As such, the semiannual excess emission report required by 40 CFR 60.7(c)-(d) and the semiannual data assessment report (DAR) required by 40 CFR Part 60 Appendix F, Procedure 1, Section 7 will address compliance with respect to the more stringent CD requirements and the AMP. These reports are attached for the January 1 to June 30, 2014 semiannual reporting period.

The relevant SO₂ standards required by the CD and AMP are as follows:

No. 8 Unit

- Per CD paragraph 11.b.i, emissions of SO₂ are not to exceed a long term limit of 1.70 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per CD paragraph 11.b.ii, emissions of SO₂ are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

Solvay USA Inc. Page 2

As discussed in the AMP, Solvay uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO₂ and % O₂) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b).

Houston #2

- Per CD paragraph 11.b.viii, emissions of SO₂ are not to exceed a long term limit of 1.80 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per CD paragraph 11.b.ii, emissions of SO₂ are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

As discussed in the AMP, Solvay uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO₂) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b)

If you have any questions or require additional information, please contact Floyd Dickerson at 713-924-1408.

Sincerely,

William McConnell

Plant Manager

Attachment(s)

cc:

Air Section Manager, TCEQ Region 12

Mr. Bob Allen, Director, Harris County Pollution Control

Mr. Arturo Blanco, Bureau Chief of Air Quality Control, Health and Human Services Department, City of Houston

Mr. Huimamshu Vyas, EPA Region 6, 1445 Ross Avenue, Suite 1200, Mailcode 6ENAT, Dallas, TX 75202-2733

EPA Region 6, New Source Review Program, 1445 Ross Avenue, Dallas, TX 75202-2733

NSPS Excess Emissions Report January 1 – June 30, 2014

Pollutant:	Sulfur Dioxide (SO ₂)
Reporting period dates:	January 1 – June 30, 2014
Emission Limitation:	Houston #8: 3.00 lbs/ton short-term, 1.70 lbs/ton long-term Houston #2: 3.00 lbs/ton short-term, 1.80 lbs/ton long-term
Address:	8615 Manchester Street, Houston, Texas 77012
Process Unit Description (Source Unit No):	Houston #8 Sulfuric Acid Unit
Monitor Manufacturer and Model No (Stack SO ₂):	Ametek Model 920
Date of Latest CEMS Certification or Audit (Stack):	June 28, 2014
CEMS span values per the AMP (Stack) (1):	Dual range: Normal: $0-500$ ppm SO_2 SSM: $0-3,600$ ppm SO_2
Process Unit Description (Source Unit No):	Houston #2 Sulfuric Acid Unit
Monitor Manufacturer and Model No (Stack SO ₂):	Ametek Model 920
Monitor Manufacturer and Model No (Converter Inlet SO ₂):	Ametek Model 920
Date of Latest CEMS Certification or Audit (Stack):	April 7, 2014
Date of Latest CEMS Certification or Audit (Converter Inlet):	April 9, 2014
CEMS span values per the AMP (Stack) (2):	Dual range: Normal: $0-500$ ppm SO_2 SSM: $0-3,600$ ppm SO_2
CEMS span values per the AMP (Converter Inlet) (2):	Single range: 0 – 15 % SO ₂

Notes:

⁽¹⁾ Refer to EPA approved Alternative Monitoring Plan for the Houston #8. (2) Refer to EPA approved Alternative Monitoring Plan for the Houston #2.

NSPS Excess Emissions Report July 1 – December 31, 2013

No. 8 Emission data summary - Long-Term Limit

. Startup/shutdown	0 hours
o. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
al duration of excess emission	0 hours
al duration of excess emissions as percent of total source operating time	0%

No. 8 Emission data summary - Short-Term Limit

a. Startup/shutdown	NA – limit does not apply during startup/shutdown
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
Total duration of excess emission	0 hours
Total duration of excess emissions as percent of total source operating time	0%

NSPS Excess Emissions Report January 1 – June 30, 2014

Houston #8 Unit Stack SO₂ Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	98.8 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	98.8 hours ⁽¹⁾
3. Total CEMS Downtime as percent of total source operating time	2.27 %

Houston #8 Unit Stack O2 Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	98.8 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	98.8 hours ⁽¹⁾
. Total CEMS Downtime as percent of total source operating time	2.27 %

⁽¹⁾ The Houston #8 Unit followed procedures specified in an EPA approved Alternative Monitoring Plan (AMP) for CEMS malfunctions. In accordance with the AMP, during CEMS malfunctions lasting more than 24 continuous hours Solvay USA Inc. generally:

Conducted sampling with hand held monitors when the stack SO₂ and O₂ CEMS malfunctioned.

NSPS Excess Emissions Report July 1 – December 31, 2013

Houston #2 Emission data summary - Long-Term Limit

1. Duration of excess emissions (as defined per CD and AMP) in reporting per	iod due to:
a. Startup/shutdown	0 hours
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total duration of excess emission	0 hours
3. Total duration of excess emissions as percent of total source operating time	0%

Houston #2 Emission data summary - Short-Term Limit

a. Startup/shutdown	NA – limit does not apply during startup/shutdown
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	2.55 hours
e. Unknown causes	0 hours
. Total duration of excess emission	2.55 hours
. Total duration of excess emissions as percent of total source operating time	3.5%

NSPS Excess Emissions Report-April 1 – June 30, 2014

Houston #2 Stack SO₂ Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	50.8 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	50.8 hours ⁽¹⁾
3. Total CEMS Downtime as percent of total source operating time	2.33 %

Houston #2 Converter Inlet (Main Gas Blower) Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	53.0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	53.0 hours ⁽¹⁾
3. Total CEMS Downtime as percent of total source operating time	2.43 %

⁽¹⁾ The Houston #2 followed procedures specified in an EPA approved Alternative Monitoring Plan (AMP) for CEMS malfunctions. In accordance with the AMP, during CEMS malfunctions lasting more than 24 continuous hours Solvay USA Inc. generally:

Conducted sampling with hand held monitors when the stack SO₂ and converter inlet SO₂ CEMS malfunctioned

NSPS Excess Emissions Report January 1 - June 30, 2014

Data Assessment Reports (DARs) per 40 CFR Part 60 Appendix F

Analyzer/	Reporting Period	A	Any out-of- control periods			
Pollutant/Units	T er lou	Type (RATA, CGA, or RAA)	Any Out-of- Control Periods?	Notes	for Calibration Drift Assessment?	
Houston #8 Stack	1Q14	RATA	No	Report enclosed	No	
SO ₂ , ppm	2Q14	CGA	No	Report enclosed	No	
Houston #8 Stack	1Q14	RATA	No	Report enclosed	No	
O ₂ , %	2Q14	CGA	No	Report enclosed	No	
Houston #2 Stack	1Q14	NA	NA	NA	NA	
SO ₂ , ppm	2Q14	RATA	No	Report enclosed	No	
Houston #2	1Q14	NA	NA	NA	NA	
Converter Inlet SO ₂ , ppm	2Q14	CGA	No	Report enclosed	No	

Describe any changes since last quarter in CEMS, process or controls:

There have been no changes in the CEMS, process, or controls since the No. 8 Unit was started on November 19, 2008.

There have been no changes in the CEMS, process, or controls since the Regen 2 was started on April 1, 2014.

******** Certification Statement for Summary Report per 40 CFR 60.7(d)**********

I certify that the information contained in this report is true, accurate, and complete.

William McCom		
Name of Responsib	ole Official	•
W	When J. h. Concel	
Signature	U	
Plant Manager		

Title Date

Texas Registered Engineering Firm F-3986
P. O. Box 890746 • Houston, Texas 77289-0746 • (281) 332-3118

AirCO/HG-0697-O/RN100220581/RP

May 07, 2014

Mr. Floyd Dickerson Environmental Manager Rhodia Inc. 8615 Manchester Houston, Texas 77012

SUBJECT:

TRANSMITTAL OF ENTECH REPORT NO. ER2014-04-105 ENTITLED "RHODIA INC., HOUSTON PLANT, VIRGIN SULFURIC ACID UNIT NO. 8 (EPN 101) OXYGEN (O₂) AND SULFUR DIOXIDE (SO₂) CONTINUOUS EMISSION MONITORING SYSTEM (CEMS) RELATIVE ACCURACY TEST AUDIT (RATA) (REGULATED ENTITY NO. RN100220581; CUSTOMER REFERENCE NO. CN600125330; TCEQ ACCOUNT ID NO. HG-0697-O;

PERMIT NO. 19282)"

Entech Engineering Inc. conducted a RATA on the Virgin Sulfuric Acid Unit No. 8 (EPN 101) O₂ and SO₂ CEMS on March 12, 2014.

Two copies and one CD of the Entech Engineering final report that documents the findings and results of this program are enclosed. Please note that the results presented in this report only relate to the items tested or the samples as received by Entech's lab; further, this report will not be reproduced, without the written approval of the client. Please contact us at our League City, Texas office if you have any questions or comments concerning the findings of this program.

Sincerely,

W. Banks Miller IV

Environmental Scientist II

Reviewed by:

Joseph Muir

Principal Environmental Scientist

Approved by:

Edward J. Pasternak

Technical Manager

Texas Registered Engineering Firm F-3986
P. O. Box 890746 • Houston, Texas 77289-0746 • (281) 332-3118

RHODIA INC.

HOUSTON PLANT

VIRGIN SULFURIC ACID UNIT NO. 8 (EPN 101)

OXYGEN (O2) AND SULFUR DIOXIDE (SO2)

CONTINUOUS EMISSION MONITORING SYSTEM (CEMS)

RELATIVE ACCURACY TEST AUDIT (RATA)

(REGULATED ENTITY NO. RN100220581; CUSTOMER REFERENCE NO. CN600125330 TCEQ ACCOUNT ID NO. HG-0697-O; PERMIT NO. 19282)

ENTECH REPORT NO. ER2014-04-105 (PAGE 1 OF 56)

PREPARED BY

ENTECH ENGINEERING INC. LEAGUE CITY, TEXAS

MARCH 12, 2014

PREPARED FOR

RHODIA INC. HOUSTON, TEXAS

SAMPLING LOCATION

VIRGIN SULFURIC ACID UNIT NO. 8 STACK (EPN 101)
RHODIA INC.
HOUSTON, HARRIS COUNTY, TEXAS

Texas Registered Engineering Firm F-3986
P. O. Box 890746 • Houston, Texas 77289-0746 • (281) 332-3118

SECTION 1.0 SUMMARY

Entech Engineering Inc. was retained by Rhodia Inc. to conduct an oxygen (O_2) and sulfur dioxide (SO_2) Continuous Emission Monitoring System (CEMS) Relative Accuracy Test Audit (RATA) at Rhodia's Virgin Sulfuric Acid Unit No. 8 in Houston, Harris County, Texas. The objective of this program was to quality assure the continuous performance of the O_2 and SO_2 CEMS according to the specifications of EPA 40 CFR, Part 60, Appendix F.

In this program, the quality assurance test, i.e. RATA was conducted according to the 40CFR60, Appendix F, Section 5.1.1 specifications following the procedures of 40CFR60, Appendix B, Performance Specification 2 and 3 for the SO₂ and O₂ CEMS, respectively. A Performance Specification (PS) test consists of two parts, a Calibration Drift (CD) Determination and a Relative Accuracy (RA) Determination; however, a RATA only requires that the RA determination be conducted. For this program, the RATA was conducted on March 12, 2014 and was coordinated by Mr. Floyd Dickerson of Rhodia Inc. TCEQ was notified of the test, but did not attend.

The Virgin Sulfuric Acid Unit No. 8 is designated in the Texas Commission on Environmental Quality (TCEQ) permit as Emission Point Number (EPN) 101. Its CEMS comprises of a Bovar/Western Research O₂/SO₂ analyzer (Model 920, Serial Number VE-920-8700-2). Flue gas samples are continuously extracted from the stack for analysis on a wet basis. During testing, operational parameters were monitored and recorded by Rhodia personnel at fifteen-minute intervals for demonstration of process conditions.

Results of the O₂ and SO₂ CEMS RATA are presented in Table 1. A comprehensive summary which includes individual test data is presented in Table 2. Test methods and equipment descriptions are presented in Section 2.0 and results and discussions are presented in Section 3.0.

Texas Registered Engineering Firm F-3986 P. O. Box 890746 • Houston, Texas 77289-0746 • (281) 332-3118

Table 1. Rhodia Inc. Houston Plant

Virgin Sulfuric Acid Unit No. 8 (EPN 101)

Oxygen(O₂) and Sulfur Dioxide (SO₂) CEMS Relative Accuracy Test Audit (RATA) Regulated Entity No. RN100220581; Customer Reference No. CN600125330 TCEQ Account ID No. HG-0697-O; Permit No. 19282 March 12, 2014

Performance Specification Test	Continuous Emission Monitoring Systems (CEMS)				
Parameters	Oxygen (O ₂)	Sulfur Dioxide (SO ₂)			
RA Test RA Allowed	Passed +/- 1.0% O ₂	Passed 20% (RM) or 10% (STD)			
RA	NA	5.89% (RM)			

(RM) - Reference Method

(STD) - Emission Standard or Performance Specification Standard

RA - Relative Accuracy Test

NA - Not Applicable



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

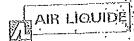
 $\binom{8}{8}$

Date: 6/28/14 Time: 11.554
Serial Number: V5-930-8700-9

Technician: Rudy Bossazo
Signature: Rudy Bossazo

				<u> </u>		
Cylinder ID number	1 ALM 045	5898	ALM 058443			
Date of Certification	7/18/2011		7/19/14			
Type of certification	EPA Protocol		EPA Protocol			
(e.g. EPA Protocol 1	5111 660 60001		1			
or CRM).			Q			
	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value	-		0 0			
C_a (ppm)	909	1970	909	1970	909	1970
CEM Response	0.0	0010	037		400	0.016
value C_m (ppm)	913	2019	927	2023	928	3019
Accuracy A (% or	. 2010	0 1600	1000	01004	209%	2,48%
ppm)	,4470	2.4870	1,10 /0	2.69%	0101/0	01.70 10

where $A = (C_m - C_a) \times 100$ C_a



Specially Gases LLC



Dual-Analyzed Callbration Standard

1,1426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281:474-8419

Protocol Gas

P.O. No. 4501452424.

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Dogimen 7, 42303145-003

14426 FAIRMONT PKWY
LA PORTE TX 77571

Customer RHODIA INC. STOREROOM

A'TTN PAUL BARNETT 8616 MANCHESTER HOUSTONIX 77913

ANALYTICAL INFORMATION Gas Type : SOZ

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.
Cylinder Number;
Cylinder Pressure***;
1970 PSIG

1,8Jul2011

17Jul2014 LAP0044990 Batch No:

Colullication Date: ..

ACCURACY**

COMPONENT SULFUR DIOXIDE *:

CERTIFIED CONCENTRATION (Moles)

11-2%

TRACEABILITY NIST and VSL

NITROGEN

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol procedures , September 1997.

HEFERENCE STANDARD.
TYPEISHM NO. EXI

EXPIRATION DATE 01/0/12016

CYLINDER NUMBER KA1,003078

CONCENTRATION 975.0 PPM

COMPONENT SULFUR DIOXIDE

INSTRUMENTATION

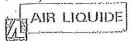
FTIR//MG-08-149

LAST DALISTATED 08Jul2011

Special Notes:

DEWYPOINT NO F. GGA 660 RDIAGRTY005

APPROVED



Air Liquide America Specially Gases LLC



COMPLIANCE CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vondor ID: A32011 .

P.O. No.: 4501462424

Customer RHODIA INC, STOREROOM

AIR LIQUIDE AMERICA SPECIALTY GASES LLG Document 11: 42303115-004

ATTN PAUL BARNETT

.. 11426 FAIRMONT PKWY

LA PORTE, TX 77571

8616 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: SO2
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

Cylinder Number; ALM058443

19Jul2011

Exp. Date:

Cylinder Number; Cylinder Pressure***;

1963 PSIG

Certification Date:

Batch No:

LAP0045081

COMPONENT SULFUR DIOXIDE *

CERTIFIED CONCENTRATION (Moles)

TRACEABILITY NIST and VSL

1,970

NITROGEN-

** Do not use when cylinder pressure is below 150 psig. ** Analytical accuracy is based on the regulrements of EPA Protocol procedures, September 1997.

REFERENCE STANDARD

TYPE/SRM NO.

NTRM 1664

EXPINATION DATE

ALM043439

CONCENTRATION.

INSTRUMENTATION NASTRUMENT/MODEL/SERIA FTIA/MOTOS 149

DEM POINT 40 F- COA 660 RUINARTYOOG



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

Date: 6/38/14 Time: 17.00 A
Serial Number: VE-920-8700-2

Cylinder ID number	ALM 036115		ALM 003590			
Date of Certification	5/20/11		5/20/11			
Type of certification	EPA Protocol		EPA Pa	otocal		
(e.g. EPA Protocol 1						
or CRM).	1					
	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value	125	974	125	274	125	274
C_a (ppm)	10-3	317	143	314	[[]	3,1
CEM Response	193	ala	122	262	190	260
value C_m (ppm)	lac	3/02	100	0,00	100	Oren
Accuracy A (% or	2 1150	4.58 %	2.45%	4.58%	4.16%	E 70 Cg
ppm)	a.70/0	4.58 10	de to to	4.5870	9.10 /0	D.JO 10

where $A = \underbrace{(C_{\underline{m}} - C_{\underline{a}})}_{C_{\underline{a}}} X \ 100$

Revised: 03/27/07



Specialty Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

· Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006 .

11426 FAIRMONT PKWY LA PORTE, TX 77671

8616 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115 Cylinder Number:

Certification Date:

20May2011

Exp. Date: Batch No:

20May 2013 LAP0041227

Cylinder Pressure***:

1934 PSIG

COMPONENT SULFUR DIOXIDE * CERTIFIED CONCENTRATION (Moles)

PPM

ACCURACY** +1-1%

+1-1-96

TRACEABILITY Direct NIST and VSL

OXYGEN NITROGEN

BALANCE

* * * Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD EXPIRATION DATE

TYPE/SRM NO.

NTRM 0260

NTRM 2350

15Ján2012 *01May2013

CYLINDER NUMBER KAL003774

K02G427

CONCENTRATION 255.5 PPM

23.50 53

COMPONENT SULFUR DIOXIDE

OXYGEN

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

FTIR/IMG-09-149

SERVOMEX/MODEL 244A/701/716

DATE LAST CALIBRATED

12May2011 25Api2011

ANALYTICAL PRINCIPLE

FTIR

PARAMAGNETIC

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Callbration Cuivo

SULFUR DIOXIDE *

Date: 13May2011 Z1 = -0.06666 R2=256,8202

23=0.15836

Response Unit: PPM R1 = 256.6585 Z2=0.11493

11=276.1549 T2=275,4641

T3=276.6497 274.1 Avg. Concentration:

R3=256,8364 PPM

R2=255,6349 72=0.15853 T3=274,7681 23=0,33653

Date: 20May2011

21=-0.32212

274.1 Avg. Concentration:

R1=256.5447.

Concentration = A.4-Bx+Cx2+Dx3+Ex4 r=9,99998E-1

Constants:

A=0.00000E+0 C=3,00000E-6

OXYGEN

Date: 19May2011 Z1=0,00000

Z3=0.00000 Avg. Concentration:

R2=0,94000

R1 = 0.94000 Z2=0.00000

Response Unit: VOLTS T1=0.61100 T2=0,61140 T3=0.61140

R3=0,94000

T2=274,6593 R3=256,5102

T1=274,2461

Response Unit: PPM

B=9,0995 JE-1 D=0.00000E+0

E=0,00000E+0

Concentration = A + Bx + Cx2 + Dx3 + Ex4

r=,9999987 Constants:

A=.000249421

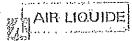
B=24.9768807

C=

Special Notest

680 CGA DEW POINT 40 F

Ramlen JR



Air Liquide America Specially Gases LLC



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-005

11426 FAIRMONT PKWY

LA PORTE, TX 77671

8615 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997. Cylinder Number:

ALM002590

Cortification Date:

20May2011

Exp. Date:

20May2013 LAP0041266

Cylinder Pressure***:

1936 PSIG

Batch No:

TRACEABILITY

COMPONENT SULFUR DIOXIDE * CERTIFIED CONCENTRATION (Moles)

ACCURACY**

+1-1%

Direct NIST and VSL

OXYGEN-

BALANCE

** Do not use when cylinder pressure is below 150 psly.

🏄 Analytical accuracy is based on the requirements of EPA Protocol Procedure 61, September 1997.

REFERENCE STANDARD

TYPE/SRM NO.

EXPIRATION DATE 16Jan 2012

NTRM 0260 NTRM 2350

NITROGEN

01May2013

CYLINDER NUMBER KAL003774

K026427

CONCENTRATION

255.5

PPM 23,50

COMPONENT SULFUR DIOXIDE

OXYGEN

Instrumentation

INSTRUMENT/MODEL/SERIAL#

FTIR////G-09-149 SERVOMEX/MODEL 244A77017716 DATE LAST CALIBRATED

12May2011 25Apr2011

ANALYTICAL PRINCIPLE

FTIR **PARAMAGNETIC**

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Triad Analysis

Gallbration Curve

SULFUR DIOXIDE *

Date: 13Mey2011 Z1 = 0.01023R2=253.7360

23=0.09222

Response Unit: PPM R1 = 253.7089

Z2=0.07108

T3=124.1083

T1=123,9645

12 = 124.0038H3=253.8609

B2=253.8925 23 = 0.12747

Z1=0.00195

Responso Unit: PPM Date: 20May2011 R1 = 253,7032Z2=0.10441

T1 = 123,9691T2 = 124.0436

T3=124.0462

H3 = 254.0720

Concentration=A+Bx+Cx2+Dx3+Ex4

r=9.99985E-1

Constants: B=9.94451E-1 0+300000E+0 C=0.00000E+0

OXYGÈÑ

Date: 19May2011

Avg. Concentration:

Avg. Concentration:

R1=0.94000

124.8

5,093

Response Unit: VOLTS T1=0.20370

Z1=0.00000 R2=0,94000 23 = 0.000000

Z2=0,00000 T3=0.20400 T2 = 0.20400R3 = 0.94000

Avg. Concentration:

124.8

D = 0.000000E + 0

E = 0.00000E + D

Concentration=A+Bx+Cx2+Dx3+Ex4

r = 9999987

Constants B=24.9768607 À=.000249421

Special Notes:

660 CGA, DEW POINT 40 F

APPROVED BY

Ramien JR



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

(8)

Date: 6/28/14 Time: 11.00 A
Serial Number: VE-920-8700-2

Technician: Roby Barraza
Signature: John Borraza

Cylinder ID number ALM 036 115		AUM002590				
Date of Certification	5/20/11		5/20/11			
Type of certification	EPA Protocol		EPA Protocol			
(e.g. EPA Protocol 1	,					
or CRM).			<u>l</u>			
	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value	٠. ٥	9	~ NO:	15.3	5.09	15.3
C_a (ppm)	5,09	15.3	5.09	15.5	5,0	10.0
CEM Response	u on	1	11 00	مسى مس	1100	15.6
value C_m (ppm)	4.87	15.5	4.89	15.5	4.89	13.6
Accuracy A (% or	11 30 637	1 306	2000	1 100	3,92%	1919
ppm)	4,32%	1,0970	3.92%	1.07 6	J, 10 10	1,10 10

where
$$A = (C_m - C_a) \times 100$$

 C_a





RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77671

· Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Multi-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customer RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Document # : 41649621-006

11426 FAIRMONT PKWY LA PORTE, TX 77671

8615 MANCHESTER HOUSTON TX 77012

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.

ALM036115

Certification Date:

20May2011

Exp. Date: Batch No:

20May2013 LAP0041227

Cylinder Number: Cylinder Pressure***!

1934 PSIG

COMPONENT

CERTIFIED CONCENTRATION (Moles)

<u>ACCURACY**</u>

TRACEABILITY

SULFUR DIOXIDE *

Direct NIST and VSL

OXYGEN NITROGEN

BALANCE

* * * Do not use when cylinder pressure is below 150 psig.

Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997,

REFERENCE STANDARD

TYPE/SRM NO.

NTRM 0260

NTRM 2360

15Ján2012 01May2013

EXPIRATION DATE

CYLINDER NUMBER

KAL003774 K026427

CONCENTRATION 255.6 PPM

23,50

COMPONENT SULFUR DIOXIDE

OXYGEN

INSTRUMENTATION INSTRUMENT/MODEL/SERIAL#

FTIR//MG-09-149

SERVOMEX/MODEL 244A)701/716

DATE LAST CALIBRATED

12May2011

ANALYTICAL PRINCIPLE

FTIR PARAMAGNETIC

26Apt2011

Response Unit: PPM

71 = 274.2461

T2=274.6593

ANALYZER READINGS

(Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

Second Tilad Analysis

Calibrátion Cuivo

SULFUR DIOXIDE*

Avg. Concentration:

Response Unit: PPM Date: 13May2011

R1=256.6585 Z1 = 0.06656

T1=276.1549 22 = 0.11493R2=256.8202 T3 = 276,6497

23=0.15836 Avg. Concentration:

274.1

12 = 275,4611

R3=258,8364

PPM

R2=255,6349 73 = 0.33653

Daio: 20May2011

Z1=-0.32212 R1=256,5447 . Z2=0.16863

R3 = 256,5102 T3=274,7681 Avg. Concentration:

Concentration = A+Bx+Cx2+Dx3+Ex4 T=9.99998E-1

Constants:

A=0.00000E+0 C=3,00000E-6

B = 9,99951E-1 D = 0.000000E + 0

E=0.00000E+0

OXYGEN

Response Unit: VOLTS Date: 19May2011

R1 =0.94000 21 = 0.00000R2=0.94000 Z2 = 0.00000Z3 = 0.00000

T2=0.61140 R3=0.94000 T3=0.61140

T1=0.61100

Concentration = A + Bx + Cx2 + Dx3 + Ex4 r=.9999987

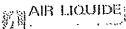
Constants: B=24,9768807 A = .000249421

Special Notest

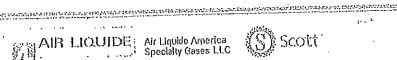
680 CGA DEW POINT 40 F

APPROVED BY

Remien JR



AIR LIQUIDE: Air Liquide America



RATA CLASS

Dual-Analyzed Calibration Standard

11426 FAIRMONT PKWY, LA PORTE, TX 77571

Phone: 800-248-1427

Fax: 281-474-8419

CERTIFICATE OF ACCURACY: Interference Free Wulti-Component EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A32011

P.O. No.: 4501416833

Customor RHODIA INC LAB

AIR LIQUIDE AMERICA SPECIALTY GASES LLC Dooument # : 41649621-005

11426 FAIRMONT PKWY LA PORTE, TX 77571

8815 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1; September, 1997.
Cylinder Number; ALM002590

20May2011

Exp. Date:

20May2013

Cylinder Number: Cylinder Pressure***:

1936 PSIG

Certification Date:

LAP0041255 Batch No:

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY** +/- 1%

TRACEABILITY

SULFUR DIOXIDE *

Direct NIST and VSL

ዕ<u>х</u>ϒઉΕΝ NITROGEN

BALANCE

*** Do not use when cylinder pressure is below 150 psig.

** Analytical accuracy is based on the requirements of EPA Protocol Procedure G1, September 1997.

REFERENCE STANDARD

TYPE/SRM NO.

NTRM 0260 NTRM 2350

15Jan2012 O1May2013

EXPINATION DATE

CYLINDER NUMBER

KAL003774 K026427

CONCENTRATION

25Apr2011

255.5 PPM 23,60 83

COMPONENT SULFUR DIOXIDE

OXYGEN

INSTRUMENTATION

INSTRUMENT/MODEL/SERIAL!

FTIR////G-09-149 SERVOMEXIMODEL 24/A/701/716 DATE LAST CALIBRATED 12May2011

ANALYTICAL PRINCIPLE

PARAMAGNETIC

ANALYZER READINGS

(Z⇒Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

First Triad Analysis

22=0.0710B

Second Trind Analysis

Calibration Curve

Concentration = A+Bx+Cx2+Dx3+Ex4

SULFUR DIOXIDE *

Date: 13May2017 21 = 0.01023R2 = 263.7350

Response Unit: PPM R1 = 253.7089

T1 = 123.9845

T2=124.0038

21=0,00195 Z2=0.10441 R2=253,8925 T3 = 124,0462

T1 = 123.9691R1 = 253.7032 T2=124,0438

Response Unit: PPM

r=9,99985E-1 Constants: B=9.94451E-1

A ≈0,00000E+0 C=0.00000E+0

73=0.09222

Avg. Concentration:

R3 = 253.8609 T3=124.1083 124.8

23=0.12747 Avg. Concentration:

Date: 20May2011

R3=254,0720 PPM

E=0,00000E+0 D=0.00000E+0

OXYGEN

Date: 19May2011 7.1 = 0.00000R2=0.94000

23≈0.00000 Avg. Concentration:

R1=0.94000 Z2 = 0.0000013 ≈ 0.20400

Response Unit: VOLTS 71=0.20370 T2 = 0.20400R3 = 0.94000 124.8

Concentration = A+Bx+Cx2+Dx3+Ex4

r = .9999987

Constants: B=24.9768607 A=.000249421

Special Notes:

660 CGA DEW POINT 40 F

APPROVED BY

Ramlen JR

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RELATIVE ACCURACY TEST AUDIT REPORT SOLVAY USA INC. SULFURIC ACID REGENERATION UNIT NO. 2 HOUSTON, TEXAS TEST DATE: 31 MARCH AND 2 APRIL 2014

Prepared for:

SOLVAY USA INC. 8715 Manchester Street Houston, TX 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way West Chester, Pennsylvania 19380

June 2014

W.O. No. 12143.075.009

1. TEST PROGRAM SUMMARY

1.1 INTRODUCTION

The Solvay USA Inc. (SOLVAY) Houston, TX facility is required to perform annual continuous emissions monitoring system (CEMS) certifications on its Sulfuric Acid Regeneration Unit No. 2 scrubber stack two range CEMS and the main gas blower CEMS. This report provides a detailed description of the monitoring procedures that were used to demonstrate compliance for each CEMS certification.

Section 1 provides a summary of the overall test program and test parameters. Section 2 provides a summary of the Relative Accuracy Test Audit (RATA) and 7-day drift test results for each CEMS tested. A description of process operations, test locations, and sampling procedures are provided in Sections 3 through 5, respectively.

Appendices A through E provide Unit 2 CEMS data and process operations data, reference method CEMS data, example calculations, equipment calibration records, and project participants, respectively.

1.2 SAMPLE PROGRAM MATRIX

Table 1-1 provides a summary of test parameters and test procedures. All CEMS RATA testing was performed on 31 March and 2 April 2014.

Table 1-1 Sample Program Matrix

Sample Sample Parameter Location		Test Method	Number of Tests	Comments
Sulfur Dioxide	Stack CEMS	EPA Method 6C	- 10	ppmv and % relative accuracy
Sulfur Dioxide	Main Gas Blower CEMS	Performance Specification 2 Alternative Method Section 16.2	2 x 3 ¹	% SO ₂ and % difference

^{1.} Two reference gas cylinders were used, each cylinder challenged the SO₂ analyzer with the known SO₂ concentration three separate times.

2. RESULTS AND DISCUSSION

The CEMS certification results are shown in Table 2-1. The test results for relative accuracy met the performance specification criteria for each CEMS tested. The SOLVAY stack SO₂ CEMS operates at two ranges, 0-500 ppm and 0-3,600 ppm. One RATA test was used to evaluate both CEMS ranges. Ten reference method test runs were conducted at the stack CEMS location. The relative accuracy was based upon nine of the ten runs. Run Nos. 1 through 9 were used in the RATA calculations. Run No. 10 was not used in the RATA calculations. All ten RATA test runs are reported in the appendices.

The test results for 7-day calibration drift represent the greater value of zero and span error percentages. These test parameters also met the performance specification criteria for each CEMS tested. Any differences between the calculated results shown in the appendices and the reported results in the summary table are due to rounding the results for presentation.

Appendix B provides detailed summaries of the relative accuracy and performance specification testing.

Table 2-1
Summary of CEMS Performance Specification Test Results

	Relative Accuracy		7-Day Calibration Drift		7 – Day Zero Drift	
Stack Analyzers	Performance Required (%)	Performance Demonstrated (%)	Performance Required (%)	Performance Demonstrated (%)	Performance Required (%)	Performance Demonstrated (%)
SO ₂	20	4.7 (Low) 5.5 (High)	≤ 2.5	0.8 (Low) 0.2 (High)	≤ 2.5	0.2 (Low) 0.03 (High)

Converter	Relative Accuracy (Alternative Method 16.2)		7-Day Calibration Drift		7 – Day Zero Drift	
Analyzer (Main Gas Blower)	Performance Required (%)	Performance Demonstrated (%)	Performance Required (%)	Performance Demonstrated (%)	Performance Required (%)	Performance Demonstrated (%)
SO ₂	15	4.2 (low conc.) 2.7 (mid conc.)	≤2.5	0.7	≤ 2.5	1.3

asking more from chemistry®

ALACIEN



Solvay USA Inc. Houston Plant

Air/Toxics & Inspection Coordination Branc' 6EN-A

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 1316)

June 6, 2014

U.S. Environmental Protection Agency Fines and Penalties Cincinnati Finance Center PO Box 979077 St. Louis, MO 63197-9000

Re:

Solvay USA Inc.

Houston Plant

8615 Manchester Street

Houston, TX 77012 Consent Agreement and Final Order in the matter of Solvay USA Inc.

Docket No. CAA-06-2014-3314

Dear Sir or Madame:

Please find check number 0005006681 for \$31,500.00 to cover the penalty assessed in Consent Agreement and Final Order in the matter of Solvay USA Inc., Docket No. CAA-06-2014-3314.

If you have any questions, please do not hesitate to contact me at (713) 924-1408.

Sincerely,

W. F. Dickerson

Environmental Manager

attachment

cc:

Carlos Flores

Enforcement Officer (6EN-AT)

Toxics Enforcement Section

Compliance Assurance and Enforcement Division

U.S EPA, Region 6

1445 Ross Avenue, Suite 1200

Dallas, TX 75202-2733

Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012



US ENVIRONMENTAL PROTECTION AGENCY FINES & PENALTIES FIFRA 0620120343 PO Box 979077 SAINT LOUIS, MO, 63197-9000 Solvay USA Inc. CN 1120 Cranbury, NJ 08512

Tel: Help Desk, 1-877-463-7645

Page 1 of 1

Check:

0005006681

Date:

06/03/2014

1008917 / US ENVIRONMENTAL PROTECTION AGENCY

Invoice Number Remarks/Description	Invoice Date	PO number	Gross amount	Discount Amount	Net amount
CR050714	05/07/2014	1900007801	31,500.00	0.00	31,500.00
	en e				
		Total :	31,500.00	0.00	31,500.00

THE ORIGINAL DOCUMENT HAS A WHITE REFLECTIVE WATERMARK ON THE BACK. HOLD AT AN ANGLE TO VIEW, DO NOT CASH IF NOT PRESENT.

S solvay

HSBC BANK, USA ONE HSBC CENTER BUFFALO NY 14203 0005006681 DATE: 06/03/2014

Solvay, USA Inc. CN 1120 Cranbury, NJ 08512

50-682 213

\$***31,500.00 US DOLLARS

PAY TO THE ORDER OF US ENVIRONMENTAL PROTECTION AGENCY

THIRTY-ONE THOUSAND FIVE HUNDRED USD ***

US ENVIRONMENTAL PROTECTION AGENCY FINES & PENALTIES FIFRA 0620120343 PO Box 979077 SAINT LOUIS, MO, 63197-9000

CAA 06-2014-3314

Salvay USA Inc.
Authorized signature

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 6 DALLAS, TEXAS

FILED 2014 MAY 21 PM 2: 35

		REGIONAL HEARING CLERK
IN THE MATTER OF:	§	DOCKET NO. CAA 06-2014-3314
IN THE MATTER OF:	8 §	DOCKET NO. CAA 00-2014-3314
SOLVAY USA INC.	§	COMPLAINT AND
	§	CONSENT AGREEMENT AND
HOUSTON, TEXAS	§	FINAL ORDER
	§	
	§	

CONSENT AGREEMENT AND FINAL ORDER

The Director, Compliance Assurance and Enforcement Division, United States

Environmental Protection Agency, Region 6 ("EPA") ("Complainant"), and SOLVAY USA Inc.

located in Houston, Texas ("Respondent" and "SOLVAY"), in the above referenced action, have agreed to resolve this matter, through issuance of this Complaint and Consent Agreement and Final Order ("Complaint" and "CAFO").

I. PRELIMINARY STATEMENT

- 1. This proceeding is the assessment of civil penalties pursuant to Section 113(d) of the Clean Air Act, as amended (CAA or the Act), 42 U.S.C. § 7413(d), and for additional terms of settlement as agreed to by Respondent. This proceeding was instituted by the issuance of a Complaint and Notice of Opportunity for Hearing ("Complaint") incorporated herein, and is simultaneously concluded by the issuance of this CAFO against Respondent pursuant to 40 C.F.R. §§ 22.13(b), 22.18(b)(2), 22.18(b)(3), and 22.34.
- This Complaint alleges that Respondent has violated the provisions governing
 Chemical Accident Prevention, and specifically the requirements to maintain training records of

employees and develop appropriate operating procedures and annually certify them, which is required by 40 C.F.R. Part 68 and Section 112(r) of the Act, 42 U.S.C. § 7412(r), at its Houston, Texas facility. Furthermore, this CAFO serves as notice pursuant to Section 113(d)(2)(A) of the Act, 42 U.S.C. § 7413(d)(2)(A), of EPA's intent to issue an order assessing penalties for this violation.

- 3. For purposes of this proceeding, Respondent admits the jurisdictional allegations of this Complaint; however, Respondent neither admits nor denies the specific factual allegations contained in this Complaint.
- 4. Respondent waives any right to contest the allegations in the CAFO and its right to appeal the Final Order set forth herein, and waives all defenses which have been raised or could have been raised to the claims set forth in the CAFO.
- 5. Compliance with all the terms and conditions of this CAFO shall only resolve Respondent's liability for federal civil penalties for the violations alleged in the CAFO.
- 6. Respondent consents to the issuance of this CAFO hereinafter recited, consents to the assessment and payment of the stated civil penalty in the amount and by the method set out in this CAFO.
- 7. Respondent shall not assert, and may not maintain, any defense or claim based upon the principles of waiver, res judicata, collateral estoppel, issue preclusion, or claim-splitting for violations not alleged in this Complaint.
- 8. Nothing in this CAFO shall be construed to prevent or limit EPA's civil and criminal authorities, or that of other Federal, State, or local agencies or departments to obtain penalties or injunctive relief under other Federal, State, or local laws or regulations.

- 9. Respondent hereby certifies that as of the date of execution of this CAFO, the Facility has corrected the violation alleged herein, and is now, to the best of its knowledge, in compliance with all the requirements of 40 C.F.R. Part 68 and Section 112(r) of the Act, 42 U.S.C. § 7412(r).
- 10. Respondent represents that the undersigned representative is fully authorized by the Party whom he or she represents to enter into the terms and conditions of this CAFO, to execute this CAFO, and to legally bind the Respondent to the terms and conditions of this CAFO.
- 11. Respondent agrees that the provisions of this CAFO shall be binding on its officers, directors, employees, agents, servants, authorized representatives, successors, and assigns, but not limited to, subsequent purchasers. Nothing in the previous sentence shall adversely affect any right of EPA under applicable law to assert successor or assignee liability against Respondent's successor or assignee, even if not owned in whole or in part, directly or indirectly, by Respondent.

II. STATUTORY AND REGULATORY BACKGROUND

- 12. Pursuant to CAA § 112(r)(7), 42 U.S.C. § 7412(r)(7), the Administrator is authorized to promulgate release prevention, detection, and correction requirements.
- 13. On June 20, 1996, the EPA promulgated a final rule known as the Chemical Accident Prevention Provisions, 40 C.F.R. Part 68, which implements Section 112(r)(7), 42 U.S.C. § 7412(r)(7), of the Act. These regulations require owners and operators of stationary sources, as defined in 40 C.F.R. § 68.3, that have more than a threshold quantity of a regulated

substance in a process no later than the latter of June 21, 1999, or the date on which a regulated substance is first present above the threshold quantity in a process, to develop, implement, and submit a RMP.

- 14. The regulations in 40 C.F.R. Part 68 set forth the requirements for the RMP that must be followed at each applicable stationary source, including the requirements regarding operating procedures and training.
- 15. Pursuant to 40 C.F.R. § 68.69(a)(3)(iii-v), "(a) The owner or operator shall develop and implement written operating procedures that provide clear instructions for safely conducting activities involved in each covered process consistent with the process safety information and shall address at least the following elements... (3) Safety and health considerations: ... (iii)

 Control measures to be taken if physical contact or airborne exposure occurs; (iv) Quality control for raw materials and control of hazardous chemical inventory levels; and, (v) Any special or unique hazards."
- 16. Pursuant to 40 C.F.R. § 68.69(c), "The operating procedures shall be reviewed as often as necessary to assure they reflect current operating practice, including changes that result from changes in process chemicals, technology, and equipment, and changes to stationary sources. The owner and operator shall certify annually that these operating procedures are current and accurate."
- 17. Pursuant to 40 C.F.R. § 68.54(a),(b): "(a) The owner or operator shall ensure that each employee presently operating a process, and each employee newly assigned to a covered process have been trained or tested competent in the operating procedures provide in § 68.52 that pertain to their duties. For those employees already operating a process on June 21, 1999, the owner or operator may certify in writing that the employee has the required knowledge, skills,

and abilities to safely carry out the duties and responsibilities as provided in the operating procedures. (b) Refresher training. Refresher training shall be provided at least every three years, and more often if necessary, to each employee operating a process to ensure that the employee understands and adheres to the current operating procedures of the process. The owner or operator, in consultation with the employees operating the process, shall determine the appropriate frequency of refresher training."

- 18. Pursuant to 40 C.F.R. § 68.71(c), "Training documentation. The owner or operator shall ascertain that each employee involved in operating a process has received and understood the training required by this paragraph. The owner or operator shall prepare a record which contains the identity of the employee, the date of training, and the means used to verify that the employee understood the training."
- 19. "Owner or operator" shall mean any person who owns, leases, operates, controls, or supervises a stationary source. 42 U.S.C. § 7412(a)(9).
- 20. "Stationary source" shall mean any buildings, structures, equipment, installations, or substance emitting stationary activities which belong to the same industrial group, which are located on one or more contiguous properties, which are under the control of the same person (or persons under common control), and from which an accidental release may occur. The term stationary source does not apply to transportation, including storage incident to transportation, of any regulated substance or any other extremely hazardous substance under the provisions of this part. A stationary source includes transportation containers used for storage not incident to transportation and transportation containers connected to equipment at a stationary source for loading or unloading. 40 C.F.R. § 68.3; CAA § 112(r)(2)(C).

- 21. "Threshold quantity" shall mean the quantity specified for regulated substances pursuant to Section 112(r)(5) of the Act, as amended in 40 C.F.R. § 68.130, Table 1, and determined to be present at a stationary source, as specified in 40 C.F.R. § 68.115. 40 C.F.R. § 68.3.
- 22. "Regulated substance" shall mean any substance listed pursuant to Section 112(r)(3) of the Act, as amended in 40 C.F.R. § 68.130. 40 C.F.R. § 68.3.
- 23. "Process" shall mean any activity involving a regulated substance including any use, storage, manufacturing, handling, or on-site movement of such substances, or combination of these activities. For the purposes of this definition, any group of vessels that are interconnected, or separate vessels that are located such that a regulated substance could be involved in a potential release, shall be considered a single process. 40 C.F.R. § 68.3.

III. FINDINGS OF FACT AND CONCLUSIONS OF LAW

- 24. Respondent is incorporated in the state of Delaware and is authorized to do business in the State of Texas.
- 25. Respondent is a "person" as that term is defined in Section 302(e) of the Act, 42 U.S.C. § 7602(e), and within the meaning of Section 113(d) of the Act, 42 U.S.C. §7413(d).
- 26. At all times relevant to this CAFO, Respondent owns and operates a chemical manufacturing facility located at 8615 Manchester Street, Houston, Texas 77012 ("Facility").
- 27. Respondent is the owner and operator of a stationary source producing, handling, or storing substances listed pursuant to CAA § 112(r)(3) or extremely hazardous substances, as published and listed in the Emergency Planning and Community Right-to-know Act of 1986 [42 U.S.C.A. § 11001 et seq.].

- 28. The Chemical Accident Prevention Provisions, 40 C.F.R. Part 68, apply to owners and operators of stationary sources that have more than a threshold quantity in a process of a substance listed pursuant to CAA § 112(r)(3).
- 29. On April 22-24, 2013, EPA conducted on onsite CAA Partial Compliance Evaluation at the Facility to verify compliance with 40 C.F.R. Part 68.
- 30. The violations were discovered from a review of the documents obtained during the inspection.

IV. VIOLATIONS

Count 1: Failure to certify annually that the required operating procedures are current and accurate (40 C.F.R. § 68.69(c)).

- 31. Respondent is subject to the Risk Management Plan ("RMP") regulations enumerated in 40 C.F.R. Part 68.
- 32. 40 C.F.R. § 68.69(c) requires that operating procedures shall be reviewed as often as necessary to assure that they reflect current operating practice, including changes that result from changes in process chemicals, technology, equipment, and changes to stationary sources. The owner or operator shall certify annually that these operating procedures are current and accurate.
- 33. From 2008 to present, the Respondent has failed on one instance to annually certify that operating procedures were current and accurate for Unit No. 8 located at the facility.
- 34. Through its failure to annually certify that required operating procedures were current and accurate, Respondent has violated 40 C.F.R. §68.69(c).

- Count 2: Failure to develop written operating procedures that provide clear instructions that include the required safety and health considerations
- 35. EPA realleges and hereby incorporates by reference Paragraphs 1-34 as referenced above.
- 36. 40 C.F.R. § 68.69(a)(3)(iii-v) requires, in relevant part, that the owner or operator of a facility shall develop and implement written operating procedures that provide clear instructions for safely conducting activities involved in each covered process consistent with the process safety information and shall address at least the following elements... (3) Safety and health considerations: ... (iii) Control measures to be taken if physical contact or airborne exposure occurs; (iv) Quality control for raw materials and control of hazardous chemical inventory levels; and, (v) Any special or unique hazards.
- 37. Through a review of Respondent's operating procedures for multiple units, it was observed that the procedures lacked information regarding the control measures to be taken if physical contact or airborne exposure occurs, quality control for raw materials and control of hazardous chemical inventory levels, and any special or unique hazards.
- 38. Through its failure to include the information listed in Paragraph 37 in its operating procedures, Respondent has violated 40 C.F.R. § 68.69(a)(3)(iii-v).
- Count 3: Failure to provide proper training documentation regarding required initial and refresher training.
- 39. EPA realleges and hereby incorporates by reference Paragraphs 1-38 as referenced above.
- 40. 40 C.F.R. § 68.71(c) requires the owner or operating of a facility to ascertain that each employee involved in operating a process has received and understood the training required by 40 C.F.R. Part 68. Further the owner or operator shall prepare a record which contains the

identity of the employee, the date of training, and the means used to verify that the employee understood the training.

- 41. After review of the training records provided for operations in the Regeneration 2, Unit No. 8, and Logistics area of the facility, it was noted that documents provided lacked the information required to determine whether each entry is an initial training or refresher training. Means to verify that the employees understood the training was not consistent, e.g. some documents were signed and acknowledged by the employee being trained, while others were not.
- 42. Through its failure to include the information listed in Paragraph 40 of this complaint in its required training documentation, respondent has violated 40 C.F.R. § 68.71(c).

V. CIVIL PENALTY AND TERMS OF SETTLEMENT

- 43. For the reasons set forth above, Respondent has agreed to pay a civil penalty which has been determined in accordance with Section 113(b)(2) of the Act, 42 U.S.C. § 7413(b)(2), which authorizes EPA to assess a civil penalty of up to twenty-five thousand dollars (\$25,000) per day for each violation of the CAA.
- 44. Upon consideration of the entire record herein, including the Findings of Fact and Conclusions of Law, which are hereby adopted and made a part hereof, and upon consideration of the size of the business, the economic impact of the penalty on the business, the violator's full compliance history and good faith efforts to comply, the duration of the violation, payment by the violator of penalties previously assessed for the same violation, the economic benefit of

¹ The Civil Penalty Inflation Adjustment Act of 1990, 28 U.S.C. § 2461, as amended by 31 U.S.C. § 3701 provides for increases in the statutory penalty provisions (\$25,000) cited in the Clean Air Act Stationary Source Civil Penalty Policy dated October 25, 1991 (CAA Penalty Policy). It provides for up to \$25,000 per day of violation for violations occurring on or before January 30, 1997; up to \$27,500 per day for each violation occurring after January 30, 1997 through March 15, 2004; up to \$32,500 per day for each violation occurring after March 15, 2004 through January 12, 2009; and up to \$37,500 per day for each violation occurring after January 12, 2009.

noncompliance, the seriousness of the violation, specific facts and equities, litigation risks, and other factors as justice may require, it is ORDERED that Respondent be assessed a civil penalty in the amount of \$31,500.

45. Within thirty (30) days of this fully executed CAFO, Respondent shall pay \$31,500 by cashier's check, certified check, or wire transfer made payable to "Treasurer, United States of America, EPA - Region 6." Payment shall be remitted in one of five (5) ways: regular U.S. Postal Service mail, to include certified mail; overnight mail; wire transfer; Automated Clearinghouse for receiving US currency; or On Line Payment. For regular U.S. Postal Service mail, U.S. Postal Service certified mail, or U.S. Postal Service express mail, the check(s) should be remitted to:

U.S. Environmental Protection Agency Fines and Penalties Cincinnati Finance Center PO Box 979077 St. Louis, MO 63197-9000

For overnight mail (non-U.S. Postal Service, e.g. FedEx), the check(s) should be remitted to:

U.S. Bank Government Lockbox 979077 U.S. EPA Fines & Penalties 1005 Convention Plaza SL-MO-C2-GL St. Louis, MO 63101 Contact: Natalie Pearson 314-418-4087 *In re* Solvay USA, Inc. Docket No. CAA-06-2014-3314

For wire transfer, the payment should be remitted to:

Federal Reserve Bank of New York

ABA: 021030004

Account Number: 68010727 SWIFT address: FRNYUS33

33 Liberty Street New York, NY 10045

Field Tag 4200 of the Fedwire message should read: "D 68010727 Environmental Protection Agency"

For Automated Clearinghouse (also known as REX or remittance express):

U.S. Treasury REX / Cashlink ACH Receiver

ABA: 051036706

Account Number: 310006, Environmental Protection Agency

CTX Format Transaction Code 22 – checking Physical location of U.S. Treasury facility:

5700 Rivertech Court Riverdale, MD 20737

Contact - Jesse White (301) 887-6548

For On Line Payment:

WWW.PAY.GOV

Enter sfo 1.1 in search field

Open form and complete required fields.

PLEASE

NOTE:

The docket number CAA 06-2014-3314 shall be clearly typed on the check to ensure proper credit. The payment shall also be accompanied by a transmittal letter and shall reference Respondent's name and address, the case name, and docket number of the administrative complaint and CAFO. Respondent's adherence to this request will ensure proper credit is given when penalties are received for the Region. Respondent shall also send a simultaneous notice of such payment, including a copy of the money order, or check, and the transmittal letter to the following:

Carlos Flores
Enforcement Officer (6EN-AT)
Toxics Enforcement Section
Compliance Assurance and Enforcement Division
U.S. EPA, Region 6
1445 Ross Avenue Suite 1200
Dallas, Texas 75202-2733;

Lorena Vaughn Region 6 Hearing Clerk (6RC-D) U.S. EPA Region 6 1445 Ross Avenue, Suite 1200 Dallas, TX 75202-2733

- 46. Respondent agrees not to claim, or attempt to claim, a federal income tax deduction or credit covering all or any part of the civil penalty paid to the United States Treasurer.
- 47. Pursuant to 31 U.S.C. § 3717 and 40 C.F.R. § 13.11, unless otherwise prohibited by law, EPA will assess interest and late payment penalties on outstanding debts owed to the United States and a charge to cover the costs of processing and handling a delinquent claim. Interest on the civil penalty assessed in this CAFO will begin to accrue thirty (30) days after the effective date of the CAFO and will be recovered by EPA on any amount of the civil penalty that is not paid by the respective due date. Interest will be assessed at the rate of the United States Treasury tax and loan rate in accordance with 40 C.F.R. § 13.11(a). Moreover, the costs of the Agency's administrative handling of overdue debts will be charged and assessed monthly throughout the period the debt is overdue. See 40 C.F.R. § 13.11(b).
- 48. EPA will also assess a fifteen dollar (\$15.00) administrative handling charge for administrative costs on unpaid penalties for the first thirty (30) day period after the payment is due and an additional fifteen dollars (\$15.00) for each subsequent thirty (30) day period that the penalty remains unpaid. In addition, a penalty charge of up to six percent per year will be assessed monthly on any portion of the debt which remains delinquent more than ninety (90) days. See 40 C.F.R. § 13.11(c). Should a penalty charge on the debt be required, it shall accrue from the first day payment is delinquent. See 31 C.F.R. § 901.9(d). Other penalties for failure to make a payment may also apply.

- 49. Pursuant to Section 113(d)(5) of the Act, 42 U.S.C. § 7413(d)(5), any person who fails to pay on a timely basis, a civil penalty ordered or assessed under this section shall be required to pay, in addition to such penalty and interest, the United States enforcement expenses, including but not limited to, attorneys fees and costs incurred by the United States for collection proceedings, and a quarterly nonpayment penalty for each quarter during which such failure to pay persists. Such nonpayment penalty shall be ten (10) percent of the aggregate amount of such person's outstanding penalties and nonpayment penalties accrued as of the beginning of each quarter.
- 50. This CAFO shall not relieve the Respondent of its obligation to comply with all applicable provisions of federal, state or local law, nor shall it be construed to be a ruling on, or determination of, any issue related to any federal, state or local permit, nor shall it be construed to constitute EPA approval of any equipment or technology installed by the Respondent in connection with any additional settlement terms undertaken pursuant to this CAFO. Nothing in this CAFO shall be construed to prohibit or prevent the federal, state, or local government from developing, implementing, and enforcing more stringent standards through rulemaking, the permit process, or as otherwise authorized or required.
- 51. This document is a "Final Order" as that term is defined in the CAA Penalty Policy for the purpose of demonstrating a history of "prior such violations."

VI. RETENTION OF ENFORCEMENT RIGHTS

52. EPA does not waive any rights or remedies available to EPA for any violations by the Respondent of federal laws, regulations, statutes, or permitting programs.

In re Solvay USA, Inc. Docket No. CAA-06-2014-3314

53. Nothing in this CAFO shall relieve Respondent of the duty to comply with all applicable provisions of the CAA.

VII. COSTS

54. Each party shall bear its own costs and attorneys fees.

In re Solvay USA, Inc.
Docket No. CAA-06-2014-XXXX
3314

IT IS SO AGREED:

FOR THE RESPONDENT:

Date: 4/30/2014

William J. Comelf

SOLVAY USA INC.

FOR THE COMPLAINANT:

Date: 5/20/14

John Blevins

Director

Compliance Assurance and Enforcement Division

FINAL ORDER

Pursuant to Section 113(d) of the Clean Air Act (Act), 42 U.S.C. § 7413(d), and the Consolidated Rules of Practice Governing the Administrative Assessment of Civil Penalties, 40 C.F.R. Part 22, the foregoing Consent Agreement is hereby ratified. This Final Order shall not in any case affect the right of EPA or the United States to pursue appropriate injunctive or other equitable relief or criminal sanctions for any violations of law. This Final Order shall resolve only those causes of action alleged in this CAFO. Nothing in this Final Order shall be construed to waive, extinguish, or otherwise affect Respondent's (or its officers, agents, servants, employees, successors, or assigns) obligation to comply with all applicable federal, state, and local statutes and regulations, including the regulations that were the subject of this action. The Respondent is ordered to comply with the terms of settlement as set forth in the Consent Agreement, including the assessment of civil penalties. In accordance with 40 C.F.R. Part 22.31(b), this Final Order shall become effective upon filing with the Regional Hearing Clerk.

Dated $\frac{5}{20/14}$

Regional Judicial Officer U.S. EPA, Region 6

CERTIFICATE OF SERVICE

I hereby certify that on the Alay of Complaint, 2014, the original and one copy of the foregoing Complaint and Consent Agreement and Final Order ("Complaint and CAFO") was hand delivered to the Regional Hearing Clerk, U.S. EPA - Region 6, 1445 Ross Avenue, Suite 1200, Dallas, Texas 75202-2733, and a true and correct copy was delivered to the following individual(s) by the method indicated below:

CERTIFIED MAIL - RETURN RECEIPT REQUESTED: 70073020000015228021

William McConnell Plant Manager SOLVAY USA, INC. Houston Facility 8615 Manchester Street Houston, Texas 77012

Lori Jackson

U.S. EPA Region 6, Dallas, Texas

441890 V3 AV/AV/EN





JAN 1 6 2015

Ant foxics & inspection Coordination Branch 6EN-A

CERTIFIED MAIL: Return Receipt Requested (7011 2970 0000 3521 0602)

January 12, 2015

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

October 1, 2014 to December 31, 2014

EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations LLC, formally Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

Mr. Robinson Page 2

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Willin Jin-Comell

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Eco Services Operations LLC Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: <u>December 31, 2014</u>

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

110000460901

Babst Calland Attorneys at Law

Michael H. Winek Attorney at Law т 412.394.6538 mwinek@babstcalland.com

January 28, 2015

Am Toxics & Inspection Coordination Branc'

Via FedEx

Chief, Environmental Enforcement Section Environment and Natural Resources Division U.S. Department of Justice Box 7611 Ben Franklin Station Washington, D.C. 20044-7611

Jan Gerro U.S. EPA Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6RCEA Dallas, TX 75202

Phillip Brooks U.S. EPA Headquarters William Jefferson Clinton Building 1200 Pennsylvania Avenue, N.W. Mailcode 2242A Washington, D.C. 20460

Himanshu Vyas U.S. EPA Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6ENAT Dallas, TX 75202

DOJ No. 90-5-2-1-08500 Consent Decree Semi-Annual Report RE: U.S. v. Rhodia Inc., USDC (N.D. Ind.), Civil Action No. 2: 07-CV-134-WCL

Ladies and Gentlemen:

In accordance with Section VII of the Consent Decree ("CD") entered in the aboveentitled matter, enclosed please find the Semi-Annual Report for the Houston, Texas facility. The Report, together with the other supporting documents enclosed, satisfies the obligation to report on certain matters under the CD within 30 days after the end of each half calendar year (see CD ¶¶ 21-23).

Chief, Environmental Enforcement Section January 28, 2015 Page 2 of 2

If you have any questions or wish to discuss this submittal, please do not hesitate to contact me.

Sincerely,

Michael H. Winek, Esq. Counsel for Eco Services Operations LLC

Enclosures

Eco Services Operations LLC - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering July 1 to December 31, 2014 Civil Action No.: 2: 07-CV-134-WCL

- 1. Effective Dates:
 - a. Houston #8 July 1, 2009
 - b. Houston #2 April 1, 2014
- Status of Construction or Compliance Measures Necessary to Meet Emissions Limits.

The plant has now completed the construction and implementation of all compliance measures necessary to meet the CD emission limits for #8 Unit. The SO₂ abatement unit was started up on November 19, 2008.

Construction has been completed and implementation of all compliance measures necessary to meet the CD emission limits for #2 Unit. The SO₂ abatement unit was started up on February 7, 2014.

The plant has completed the implementation of all compliance measures necessary to meet the CD emission limits.

- 3. Compliance Issues and Proposed or Implemented Solutions
 - (a) Houston #8 Long-Term SO₂ Limit of 1.70 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #8 unit operated below the permitted 1.70 lbs. SO₂/ton of acid produced from January 1, 2014 to December 31, 2014.
 - (b) Houston #8 Short-term SO₂ limit of 3.00 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.
 - (c) Houston #2 Long-Term SO₂ Limit of 1.80 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. The start of the Long-Term Limit compliance period is April 1, 2014.
 - (d) Houston #2 Short-term SO₂ limit of 3.00 lbs./ton The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period.

Eco Services Operations LLC - Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering July 1 to December 31, 2014 Civil Action No.: 2: 07-CV-134-WCL

(e) During the reporting period, the plant has not encountered any problems, and does not anticipate encountering any problems, with any of the conditions of the Consent Decree or any applicable permits or any other event affecting the plant's performance under the Consent Decree.

4. Status of Permit Applications

Houston Title V air permit O-03049 was approved on June 28, 2012. Requirements for compliance with 40 CFR Part 60.83(a)(1) (sulfuric acid mist) Houston #2 and compliance with the Consent Decree SO₂ emission rates for Houston #2 have been included as conditions in the Title V air permit.

Operation and Maintenance Work

The plant does not currently have any operation or maintenance work pending as a result of any of the conditions of the Consent Decree.

5. Reports to Agencies

Eco Services has installed a dual range SO₂ and a new O₂ CEMS for the Houston #8 in 2008. The SO₂ and O₂ CEMS monitor and record the 3-hour arithmetic average SO₂ emission rate in units of lbs. SO₂ per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

Cylinder gas audits were conducted on the Houston #8 stack SO₂ and O₂ CEMS on September 15, 2014 and October 10, 2014. The CEMS passed these tests.

Eco Services has installed a dual range stack SO₂ CEMS and a new main gas blower SO₂ CEMS for the Houston #2 in 2014. The SO₂ CEMS monitor and record the 3-hour arithmetic average SO₂ emission rate in units of lbs. SO₂ per ton 100% acid produced.

The CEMS were certified and calibrated, and has been maintained and operated in accordance with the applicable requirements of 40 CFR 60.11, 60.13, Part 60, Appendix B Performance Specification 2, and Par 60 Appendix F Procedure 1.

Cylinder gas audits were conducted on the Houston #2 stack SO₂ CEMS on August 29, 2014 and on January 16, 2015. Cylinder gas audits were conducted on the Houston #2 main gas blower SO₂ CEMS on August 29, 2014 and January 20, 2015. The CEMS passed the gas audit tests.

Eco Services Operations LLC - Houston #8 and #2 Plants Consent Decree Semi-Annual Report for Period Covering July 1 to December 31, 2014

Civil Action No.: 2: 07-CV-134-WCL

The plant submitted Excess Emission Reports for SO₂ per 40 CFR 60.7(c)-(d) and Data Assessment Reports for SO₂ CEMS per 40 CFR Part 60, Appendix F for the first and second calendar quarters of 2014 to the US Environmental Protection Agency (USEPA) and TCEQ.

A copy of the semiannual report is attached which includes the results of the RATA and cylinder gas audits.

I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and their attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

Name/Position: William McConnell/Plant Manager of Baytown and Houston Plants

Signature: Millin Jhr Eschu

Date: 1/21/2015



Eco Services Operations LLC Houston Plant

CERTIFIED MAIL; RETURN RECEIPT REQUESTED: (7011 2000 0001 4575 0845)

January 21, 2015

Texas Commission on Environmental Quality Office of Permitting, Remediation and Registration Air Permits Division, MC-163 P.O. Box 13087 Austin, Texas 78711-3087

Subject:

Eco Services Operations LLC (CN604683482)

Houston Plant (RN100220581)

Consent Decree (Civil Action No. 2:07CV134 WL)

Air Permit 19282 and PSD-TX-1081 Air Permit 4802 and PSD-TX-1260

Excess Emission Report for SO₂ per 40 CFR 60.7(c)-(d)

Data Assessment Report for SO₂ and O₂ CEMs per 40 CFR Part 60, Appendix F

Dear Sir or Madam:

In accordance with the Consent Decree referenced above, the Eco Services Operations LLC (Eco Services), formally Solvay USA Inc. and Rhodia Inc., Houston No. 8 became subject to 40 CFR Part 60 Subpart H, Standards of Performance for Sulfuric Acid Plants on November 19, 2008 and Houston Regen 2 became subject on April 1, 2014. Further, the Consent Decree specifies a SO₂ emission standard that is more stringent than Subpart H and also incorporates an EPA-approved Alternative Monitoring Plan (AMP). As such, the semiannual excess emission report required by 40 CFR 60.7(c)-(d) and the semiannual data assessment report (DAR) required by 40 CFR Part 60 Appendix F, Procedure 1, Section 7 will address compliance with respect to the more stringent CD requirements and the AMP. These reports are attached for the July 1 to December 31, 2014 semiannual reporting period.

The relevant SO₂ standards required by the CD and AMP are as follows:

No. 8 Unit

- Per CD paragraph 11.b.i, emissions of SO₂ are not to exceed a long term limit of 1.70 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per CD paragraph 11.b.ii, emissions of SO₂ are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

Eco Services Operations LLC Page 2

As discussed in the AMP, Eco Services uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO₂ and % O₂) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b).

Houston # 2

- Per CD paragraph 11.b.viii, emissions of SO₂ are not to exceed a long term limit of 1.80 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per CD paragraph 11.b.ii, emissions of SO₂ are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

As discussed in the AMP, Eco Services uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO₂) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b)

If you have any questions or require additional information, please contact Floyd Dickerson at 713-924-1408.

Sincerely,

William McConnell Plant Manager

Attachment(s)

cc:

Air Section Manager, TCEQ Region 12

miller of . Milouly

Mr. Bob Allen, Director, Harris County Pollution Control

Mr. Arturo Blanco, Bureau Chief of Air Quality Control, Health and Human Services Department, City of Houston

Mr. Huimamshu Vyas, EPA Region 6, 1445 Ross Avenue, Suite 1200, Mailcode 6ENAT, Dallas, TX 75202-2733

EPA Region 6, New Source Review Program, 1445 Ross Avenue, Dallas, TX 75202-2733

General Information:

Pollutant:	Sulfur Dioxide (SO ₂)
Reporting period dates:	July 1 – December 31, 2014
Emission Limitation:	Houston #8: 3.00 lbs/ton short-term, 1.70 lbs/ton long-term Houston #2: 3.00 lbs/ton short-term, 1.80 lbs/ton long-term
Address:	8615 Manchester Street, Houston, Texas 77012
Process Unit Description (Source Unit No):	Houston #8 Sulfuric Acid Unit
Monitor Manufacturer and Model No (Stack SO ₂):	Ametek Model 920
Date of Latest CEMS Certification or Audit (Stack):	October 10, 2014
CEMS span values per the AMP (Stack) (1):	Dual range: Normal: 0 - 500 ppm SO ₂ SSM: 0 - 3,600 ppm SO ₂
Process Unit Description (Source Unit No):	Houston #2 Sulfuric Acid Unit
Monitor Manufacturer and Model No (Stack SO ₂):	Ametek Model 920
Monitor Manufacturer and Model No (Converter Inlet SO ₂):	Ametek Model 920
Date of Latest CEMS Certification or Audit (Stack):	January 16, 2015
Date of Latest CEMS Certification or Audit (Converter Inlet):	January 20, 2015
CEMS span values per the AMP (Stack) (2):	Dual range: Normal: 0 - 500 ppm SO ₂ SSM: 0 - 3,600 ppm SO ₂
CEMS span values per the AMP (Converter Inlet) (2):	Single range: 0 − 15 % SO ₂

Notes:

⁽¹⁾ Refer to EPA approved Alternative Monitoring Plan for the Houston #8.
(2) Refer to EPA approved Alternative Monitoring Plan for the Houston #2.

No. 8 Emission data summary - Long-Term Limit

. Startup/shutdown	0 hours
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
tal duration of excess emission	0 hours
tal duration of excess emissions as percent of total source operating time	0%

No. 8 Emission data summary - Short-Term Limit

a. Startup/shutdown	NA – limit does not apply during startup/shutdown
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
Total duration of excess emission	0 hours
Total duration of excess emissions as percent of total source operating time	0%

Houston #8 Unit Stack SO₂ Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	102.6 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	102.6 hours ⁽¹⁾
3. Total CEMS Downtime as percent of total source operating time	2.32 %

Houston #8 Unit Stack O2 Analyzer

1. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	102.6 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	102.6 hours (i)
3. Total CEMS Downtime as percent of total source operating time	2.32 %

The Houston #8 Unit followed procedures specified in an EPA approved Alternative Monitoring Plan (AMP) for CEMS malfunctions. In accordance with the AMP, during CEMS malfunctions lasting more than 24 continuous hours Eco Services generally:

• Conducted sampling with hand held monitors when the stack SO₂ and O₂ CEMS malfunctioned.

Houston #2 Emission data summary - Long-Term Limit

. Startup/shutdown	0 hours
Control equipment problems	0 hours
. Process problems	0 hours
. Other known causes	0 hours
Unknown causes	0 hours
l duration of excess emission	0 hours
al duration of excess emissions as percent of total source operating time	0%

Houston #2 Emission data summary - Short-Term Limit

a. Startup/shutdown	NA – limit does not apply during startup/shutdown
b. Control equipment problems	0 hours
c. Process problems	0 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
otal duration of excess emission	0 hours
otal duration of excess emissions as percent of total source operating time	0%

Houston #2 Stack SO₂ Analyzer

1. CEMS downtime in reporting period due to:	-
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	102.6 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	102.6 hours ⁽¹⁾
3. Total CEMS Downtime as percent of total source operating time	2.32 %

Houston #2 Converter Inlet (Main Gas Blower) Analyzer

. CEMS downtime in reporting period due to:	
a. Monitor equipment malfunctions	0 hours
b. Non-Monitor equipment malfunctions	0 hours
c. Quality assurance calibration	102.6 hours
d. Other known causes	0 hours
e. Unknown causes	0 hours
2. Total CEMS Downtime	102.6 hours ⁽¹⁾
3. Total CBMS Downtime as percent of total source operating time	2,32 %

⁽¹⁾ The Houston #2 followed procedures specified in an EPA approved Alternative Monitoring Plan (AMP) for CEMS malfunctions. In accordance with the AMP, during CEMS malfunctions lasting more than 24 continuous hours Eco Services generally:

Conducted sampling with hand held monitors when the stack SO₂ and converter inlet SO₂ CEMS malfunctioned

NSPS Excess Emissions Report July 1 - December 31, 2014

Data Assessment Reports (DARs) per 40 CFR Part 60 Appendix F

Analyzer/	Reporting	Ac	ccuracy Assessme	ent	Any out-of- control periods	
Pollutant/Units	Period	Type (RATA, CGA, or RAA)	Any Out-of- Control Periods?	Notes	for Calibration Drift Assessment?	
Houston #8 Stack	3Q14	CGA	No	Report enclosed	No	
SO ₂ , ppm	4Q14	CGA	No	Report enclosed	No	
Houston #8 Stack	3Q14	CGA	No	Report enclosed	No	
O ₂ , %	4Q14	CGA	No	Report enclosed	No	
Houston #2 Stack	3Q14	CGA	No	Report enclosed	No	
SO ₂ , ppm	4Q14	CGA	No	Report enclosed	No	
Houston #2	3Q14	CGA	No	Report enclosed	No	
Converter Inlet SO ₂ , ppm	4Q14	CGA.	No	Report enclosed	No	

Describe any changes since last quarter in CEMS, process or controls:

There have been no changes in the CEMS, process, or controls since the No. 8 Unit was started on November 19, 2008.

There have been no changes in the CEMS, process, or controls since the Regen 2 was started on April 1, 2014.

******** Certification Statement for Summary Report per 40 CFR 60.7(d)**********
I certify that the information contained in this report is true, accurate, and complete.
William McConnell Name of Responsible Official

Signature	•	
Plant Manager Title		

Date

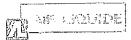


Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

2

Unit Number (Circle One):

Cylinder ID number	ALM009085		CC 58 452			
Date of Certification	10/23/1	3	/0/92	113		
Type of certification (e.g. EPA Protocol 1 or CRM).	EPA	o(EPA	مره (
	Tri	al 1	Tria	Trial 2		al 3
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1.	Audit Point 2
Certified audit value C_a (ppm)	127	980	127	980	127	980
CEM Response value C_m (ppm)	130	279	114	971	119	279
Accuracy A (% or ppm)	2.362	-,357	-10,236	-3,214	-6.299	-,357





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

883? DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: ELA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8332 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document #: 52372052-004 Folio #:RDIAQRTY003

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/631; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number: Cylinder Pressure: ALM009085

Certification Date:

230ct2013

Exp. Date:

240ct2021

2000 PSIG

SB00079450

Batch No:

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles)

PPM

ACCURACY (ABSOLUTE / RELATIVE) 1.0 PPM 0.8

BALANCE

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 100.4000 PPM

0,8000

UNCERTAINTY PPM

CYLINDER KAL003572

FTIR

TYPE/SRM SAMPLE

NTRM 1694

EXP. DATE

24Jan2018

ANALYTICAL METHOD

1st Analysis: 150ct2013

COMPONENT

SULFUR DIGXILE

INS'TRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCE TRATION

126.B PPM 25Sep2013

2nd Analysis: 230ct2013

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

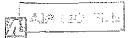
126.9 PPM

Special Notes:

125.00 PFLI 302 (100-150 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013 AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document # : 52372052-003

Folio #:RDIAQRTY004

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC58452

Certification Date:

220ct2013

Exp. Date:

230ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079448

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY (ABSOLUTE / RELATIVE)

SULFUR DIOXIDE NITROGEN

280

PPM 2.

PPM BALANCE

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 255.3000 PPM

UNCERTAINTY 2.0000

CYLINDER AAL072952

TYPE/SRM SAMPLE

NTRM 0260

EXP. DATE 20May2016

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MI(S-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION 279.6 PPM

25Sep2013

2nd Analysis: 220ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

280.6 PPM

Special Notes:

275.00 PPM SO2 (250-300 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN

1 of 1 Page



Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

(8)

Date: 9/15/14 Time: 3:10 p
Serial Number: VE-990-8700-3

Technician: Zah, Bastaze.
Signature: A Be

Cylinder ID number	ALMOI	ALM 015638		ALM 049351		
Date of Certification	10/9A	13	10/21	113		
Type of certification	EPA	•	EPA			•
(e.g. EPA Protocol 1 or CRM).	Proto	col	Proto	col		<u> </u>
OI CICIVI).	Trial 1		Tri	Trial 2		al 3
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	906	1990	906	1990	906	1990
CEM Response value C_m (ppm)	914	2075	919	9093	918	६६०६
Accuracy A (% or ppm)	,883	1.759	1.435	1,658	1,325	1,068

where $A = \frac{(C_m - C_a)}{C_a} \times 100$





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protogol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-002

Folio #:RDIAQRTY005

Customer RHODIA INC: STOREROOM

8615 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: SO2, BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

ALM015638

Certification Date:

220ct2013

Exp. Date:

230ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079446

COMPONENT SULFUR DIOXIDE CERTIFIED CONCENTRATION (Moles)

PPM **BALANCE** ACCURACY (ABSOLUTE / RELATIVE)

PPM / 0.8

NITROGEN

TRACEABILITY REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 975.0000

UNCERTAINTY 7.0000 PPM

CYLINDER KAL003179

TYPE/SRM SAMPLE

NTRM 1662

EXP. DATE

01Jun2016

ANALYTICAL METHOD

1st Analysis: 140ct2013

INSTRUMENT

MKS-FTIR/2030/001785245

906

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

906.G

2nd Analysis: 220ct2013

COMPONENT

COMPONENT

SULFUR DIOXIDE

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

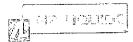
906.3 PPM

Special Notes:

900.00 PPM SO2 (800-1000 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN





COMPLIANCE CLASS

Guaranteea +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926, 8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-001 Folio #:RDIAQRTY006

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 us

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

ALM049351

Certification Date:

210ct2013

Exp. Date:

220ct2021

Cylinder Pressure:

2000 PSIG

SB00079337 Batch No:

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles) 1,990

BALANCE

ACCURACY (ABSOLUTE / RELATIVE) PPM 12.

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 2402.0000 PPM

14,0000

UNCERTAINTY CYLINDER ALM063514 PPM

TYPE/SRM SAMPLE

NTRM 1664

EXP. DATE

03Nav2017

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION 1991. PPM

FTIR

FOR

2nd Analysis: 210ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

1991. PPM

Special Notes:

1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN 1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN EPA CEMS PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN



Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

Unit Number (Circle One):

2

Date: 9/15/14

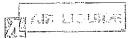
Time: 3'.059

al Number: V6-920-8700-3 Sign

Technician: fully Dorrollar Signature:

				_ _		
Cylinder ID number	CL 515	15		<u> </u>		
Date of Certification	10/25/1	3	10/23	[13		
Type of certification	EPA	Ç	EPA			•
(e.g. EPA Protocol 1 or CRM).	Bloton	_ 	Prote			
	Tri	Trial 1		Trial 2		al 3
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	5.07	15	5.07	15	5.07	15
CEM Response value C_m (ppm)	5.02	15.2	5.03	15,2	5.64	15,2
Accuracy A (% or ppm)	-,986	1.333	-,789	1,333	- 586	<i>1,33</i> 3

where $A = (C_m - C_a) \times 100$ C_a





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926 8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document #: 52372226-001 Folio #:RDIA018

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: O2,BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC51515

Certification Date:

250ct2013

Exp. Date:

260ct2021

Cylinder Pressure:

2000 PSIG

SB00079703 Batch No:

NTRM 2659

COMPONENT OXYGEN NITROGEN

CERTIFIED CONCENTRATION (Moles) 15.0

BALANCE

ACCURACY (ABSOLUTE / RELATIVE) 0.11

TRACEABILITY

REFERENCE STANDARD

COMPONENT OXYGEN

CONCENTRATION 20.9500 %

UNCERTAINTY 0.1300

CYLINDER KO16558

TYPE/SRM SAMPLE

EXP. DATE 14ivlay2018

ANALYTICAL METHOD

1st Analysis: 25Oct2013

COMPONENT OXYGEN

INSTRUMENT HP/5890F/3336A60154 ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

14.97 250ct2013

15% (+1-5%) OXYGEN, BALANCE NITROGEN EPA PROTOCOL - written certs & tags Rhodia Special Notes: APPROVED BY:



Quarterly Cylinder Gas Audit Checklist

Stack SO2 Analyzer
Tail Gas Scrubber

Unit Number (Circle One):

2

8

Date: 3/29/14 Time: 10:43 AW1
Serial Number: 2Y-980-10628-1

Technician: Andrew Kovas / F. Cortes Signature:

Cylinder ID number	CC5843	<u> </u>	ALMOO	9085		
Date of Certification	10/22/3	2013	10/23/	2014		
Type of certification (c.g. EPA Protocol I) or CRM'.	EPA Pro		EPA Pro			
	Trial I		Tria	Trial 2		al 3
	Audit	Audit	Audit	Audit	Audit	Audit
<u> </u>	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value		280	127	230	127	280
C_a (ppm)	127	800	100 1	200	10/1	300
CEM Response	135	284	130	a84	129	283
value Cn (ppm)				•		
: Accuracy A (% or ppm	6.299%	1.429%	2,362%	1,439%	1.575%	1.071%

where $\Delta = (C_{\underline{m}} + C_{\underline{n}}) \times 100$ $C_{\underline{n}}$





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013 AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document #: 52372052-003 Folio #:RDIAQRTY004 ·

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;
Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC58452

Certification Date:

220ct2013

Exp. Date:

230ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079448

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles) 280

PPM

ACCURACY (ABSOLUTE / RELATIVE) PPM / 0.7 %

BALANCE

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 255.3000

UNCERTAINTY 2,0000 PPM

CYLINDER AAL072952 TYPE/SRM SAMPLE NTRM 0260

EXP. DATE 20May2016

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

25Sep2013 279.6

2nd Analysis: 220ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

25Sep2013 280.6

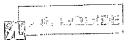
Special Notes:

275.00 PPM SO2 (250-300 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN

1 of 1 Page





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8833 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EFA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

2332 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document #: 52372052-004 Folio #:RDIAORTY003

Customer RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: SO2, BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

ALM009085

Certification Date:

230ct2013

Exp. Date:

240ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079450

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles)

1.0

NTRM 1694

ACCURACY (ABSOLUTE / RELATIVE)

PPM **BALANCE**

TRACEABILITY REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 100.4000 PPM

UNCERTAINTY 0.8000

CYLINDER_ KAL003572

TYPE/SRM SAMPLE

EXP. DATE

24Jan2018

ANALYTICAL METHOD

1st Analysis: 150ct2013

COMPONENT SULFUR DIOXINE INS TRUMENT MKS-FTIR/2030/001785245 ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION 126.8 PPM

25Sep 2013

2nd Analysis: 230ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

126.9 PPM

125.00 PF% 302 (100-150 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA Special Notes: APPROVED BY:

THUAN TRAN



Quarterly Cylinder Gas Audit Checklist

Stack SO₂ Analyzer
Tail Gas Scrubbur

Unit Number (Circle One):

(27)

8

Date: 8/29/14 Time: 10:43 AM
Serial Number: 21-920-10628-1

Technician: A. Kovac / F. Coches.
Signature: The Course

Cylinder ID number	ALMO15	ALM015638		ALM049351		
Date of Certification		10/22/2013		10/21/2013		
Type of certification (e.g. EPA Protocol I or CRM).	EPA Pre		EPA Prod			• "
	Trial 1		Tria	Trial 2		al 3
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	906	1990	906	1990	906	1990
CEM Response value C_m (ppm)	169	1980	727	1978	927	1980
Accuracy A (% or ppm)	1.656%	-0,503%	2.318%	-0.6032	2,318%	-0,503%

where A =
$$(\underline{C_m - C_n}) \times 100$$

 C_n





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-002

Folio #:RDIAQRTY005

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012

US

ANALYTICAL INFORMATION Gas Type: SO2,BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;

Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number: Cylinder Pressure: ALM015638 2000 PSIG

Certification Date:

220ct2013

Exp. Date: Batch No:

230ct2021

SB00079446

COMPONENT SULFUR DIOXIDE CERTIFIED CONCENTRATION (Moles)

ACCURACY (ABSOLUTE / RELATIVE)

NITROGEN

906

PPM

BALANCE

0.8

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 975.0000

UNCERTAINTY 7,0000

CYLINDER KAL003179

FTIR

FTIR

TYPE/SRM SAMPLE

NTRM 1662

EXP, DATE

01Jun2016

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

906.G PPM

2nd Analysis: 220ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

906.3 PPM

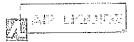
Special Notes:

900.00 PPM SO2 (800-1000 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN

- --- 1/1 (1. 1 / fr T fr)





COMPLIANCE CLASS

Guaranteeá +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document # : 52372052-001 Folio #:RDIAQRTY006

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number: Cylinder Pressure: ALM049351 2000 PSIG

Certification Date:

210ct2013

220ct2021 Exp. Date:

SB00079337 Batch No:

COMPONENT

CERTIFIED CONCENTRATION (Moles)

ACCURACY (ABSOLUTE / RELATIVE) PPM 12.

SULFUR DIOXIDE NITROGEN

PPM 1,990

BALANCE

TRACEABILITY REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 2402.0000

PPM

UNCERTAINTY 14,0000 PPM

TYPE/SRM SAMPLE CYLINDER NTRM 1664 ALM063514

EXP. DATE 03Nnv2017

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT MKS-FTIR/2030/001785245 ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

25Sep2013 1991. PPM

2nd Analysis: 210ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE CALIBRATED

25Sep2013

CONCENTRATION

1991. PPM

Special Notes:

1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN 1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN EPA. CEMS PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN



Quarterly Cylinder Gas Audit Checklist MGB SO₂ Analyzer

Date: 9/25/2014 Time: 2:54 PM Technician: Marcus AG:
Serial Number: 4338

Signature: 22

Cylinder ID number	CC 191	6449	ALMOY			
Date of Certification		12013	07 001	2013		
Type of certification (e.g. EPA Protocol 1	EPA Pa	otocol 1	EPA P.	-stocal l		, professional control
or CRM).	. Trial 1		Tria	Trial 2		al 3
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	3.75	8.24	3.75	8.24	3.75	8.24
CEM Response	3.80	3.24	3.75	8.13	3.61	7.99
value C_m (ppm) Accuracy A (% or ppm)	1.333	D	0	1.353	-3.733	-3.033

Revised: 10/13/14

where $A = (C_m - C_n) \times 100$ C_n





Single-Certified Calibration Standard

1290 COMBERMERE STREET, TROY, MI 48083

Phone: 248-589-2950 Fax: 248-589-2134

Certified Master Class Calibration Standard **CERTIFICATE OF ACCURACY:**

الک: '

Product Information Document #: 52384839-002 Item No.: M374501-P-30AL P.O. No.: 4501864926

Cylinder Number: ALMO46889

Cylinder Size: 30AL
Certification Date: 07Oct2013
Expiration Date: 08Oct2016
Lot Number: TRO0094111

CERTIFIED CONCENTRATION

Component Name,

SULFUR DIOXIDE, NITROGEN

TRACEABILITY

Traceable To

Scott Reference Standard

Customer

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON, TX 77012

Concentration (Moles)

Accuracy (+/-%)

3.75

BALANCE

APPROVED BY:

CERTIFIED MASTER CLASS



Air Liquide America Specialty Gases LLC



Single-Certified Calibration Standard

1290 COMBERMERE STREET, TROY, MI 48083

Phone: 248-589-2950 Fax: 248-589-2134

Certified Waster Class Calibration Standard CERTIFICATE OF ACCURACY:

Product Information
Document #: 52384839-003
Item No.: M374501-P-30AL
P.O. No.: 4501864926

Cylinder Number: CC196449 Cylinder Size: 30AL Certification Date: 070ct2013 Expiration Date: 080ct2016 Lot Number: TR00094123 Customer

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON, TX 77012 US

CERTIFIED CONCENTRATION

Component Name

SULFUR DIOXIDE NITROGEN

Concentration (Moles)

Accuracy (+1-%) 2

8.24

BALANCE

TRACEABILITY

Traceable To

Scott Reference Standard

	•		
APPROVED BY:	The state of the s	DATE:	



Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

High

Revised: 03/27/07

Unit Number (Circle One):

2

8

Date: 10/9/14 Time: 11:50 pm Serial Number: VE-920 - 8700-2

Technician: Envigue Nie to Signature: Envigue Mie to

						A
Cylinder ID number	ALM 15	ALM 15638		9351		V
Date of Certification	22 OC	T. 2013	21 OC	1. 2013		
Type of certification	EPA	+	EPA			
(e.g. EPA Protocol 1 or CRM).	1650	PSI	1150	PSI	<u> </u>	
	Tri	al 1	Tri	Trial 2		al 3
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	906	1990	906	1990	906	1990
CEM Response	909	1001	915	2010	916	1999
value C_m (ppm)	10 1	2001	110	0010	TIVE	1111
Accuracy A (% or	0,331	0,55.3	0.993	1,005	1.104	0.452
ppm)	الديا	<u> </u>	V. 112	1,00	1,101	0, 100

where $A = (C_m - C_a) \times 100$





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013 AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-002

Folio #:RDIAQRTY005

Customer RHODIA INC. STOREROOM 8615 MANCHESTER

HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: SO2,BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

ALM015638

Certification Date:

220ct2013

Exp. Date:

230ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079446

COMPONENT SULFUR DIOXIDE

CERTIFIED CONCENTRATION (Moles)

906 PPM **BALANCE** ACCURACY (ABSOLUTE / RELATIVE)

PPM / 0.8

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE

NITROGEN

CONCENTRATION 975,0000 PPM

UNCERTAINTY 7,0000 PPM

CYLINDER KAL003179

TYPE/SRM SAMPLE

NTRM 1662

EXP. DATE 01Jun2016

ANALYTICAL METHOD

1st Analysis:

140ct2013

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

906.C PPM

2nd Analysis: 220ct2013

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

25Sep2013

CONCENTRATION

25Sep2013 906.3 PPM

Special Notes:

900.00 PPM SO2 (800-1000 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA - myllinifisher

APPROVED BY:

THUAN TRAN





COMPLIANCE CLASS

Guaranteeá +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-001 Folio #:RDIAQRTY006

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number: .

ALM049351

Certification Date:

210ct2013

Exp. Date:

220ct2021

Cylinder Pressure:

2000 PSIG

Batch No:

SB00079337

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles) 1,990

PPM

ACCURACY (ABSOLUTE / RELATIVE) PPM / 0,6 12.

BALANCE

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 2402.0000 PPM

14.0000

CYLINDER UNCERTAINTY ALM063514 TYPE/SRM SAMPLE NTRM 1664

EXP. DATE 03Nov2017

ANALYTICAL METHOD

1st Analysis:

140ct2013

COMPONENT SULFUR DIOXIDE

INSTRUMENT MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

25Sep2013 1991. PPM

2nd Analysis: 210ct2013

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep 2013

CONCENTRATION

1991. PPM

Special Notes:

1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN 1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN EPA CEMS PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:



Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

LOW

Revised: 03/27/07

Unit Number (Circle One):

2



Date: 10/10/14 Time: 12:47 am
Serial Number: VE-920-8700-2

Technician: Envioue Nieta Signature: Envioue Nieta

						1)
Cylinder ID number	ALMOD'	ALM009085		CU58452		
Date of Certification	23 007	. 2013	22,00	T. 2013		
Type of certification (e.g. EPA Protocol 1	EPA	1		EPA		
or CRM).	1480	PSI	1500	PSI		i
	Tr	ial 1	Tri	Trial 2		al 3
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	127	280	127	280	127	280
CEM Response value C_m (ppm)	129	283	127	280	126	279
Accuracy A (% or ppm)	1.575	1.071	0,0	0.0	-0,787	-0,357

where A =
$$(C_m - C_n)$$
 X 100 C_n





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

883? DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EFA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8332 DICE ROAD SANTA FE SPRINGS, CA 90670-2516 Document #: 52372052-004 Folio #:RDIAQRTY003

RHODIA INC. STOREROOM **8615 MANCHESTER** HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

ALM009085

Certification Date:

Exp. Date:

240ct2021

Cylinder Pressure:

2000 PSIG

230ct2013

Batch No:

SB00079450

COMPONENT SULFUR DIOXIDE

CERTIFIED CONCENTRATION (Moles)

PPM

BALANCE

ACCURACY (ABSOLUTE / RELATIVE) 0.8 1.0 PPM

TRACEABILITY

SULFUR DIOXIDE

NITROGEN

REFERENCE STANDARD COMPONENT

CONCENTRATION 100,4000 PPM

127

UNCERTAINTY 0.8000 PPM

CYLINDER KAL003572

TYPE/SRM SAMPLE

NTRM 1694

EXP. DATE 24Jan2018

ANALYTICAL METHOD

1st Analysis: 150ct2013

COMPONENT SULFUR DIOX:LE

INSTRUMENT MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE FTIR

CALIBRATED 25Sep2013

CONCENTRATION 126.8 PPM

2nd Analysis: 230ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

FT18

CALIBRATED 25Sep2013

CONCENTRATION

126.9 PPM

Special Notes:

125.00 PF.: 1 302 (100-150 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN

Paga 1 of 1





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926 8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-003 Folia #:RDIAQRTY004

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC58452

Certification Date:

220ct2013

Exp. Date:

230ct2021

Cylinder Pressure:

2000 PSIG

Batch No: SB00079448

COMPONENT SULFUR DIOXIDE

CERTIFIED CONCENTRATION (Moles)

ACCURACY (ABSOLUTE / RELATIVE)

NITROGEN

280

PPM BALANCE

0.7 % PPM

TRACEABILITY REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 255.3000 PPM

UNCERTAINTY PPM 2.0000

CYLINDER AAL072952

FTIR

ETIR

TYPE/SRM SAMPLE NTRM 0260

EXP. DATE 20May2016

ANALYTICAL METHOD

1st Analysis:

140ct2013

COMPONENT SULFUR DIOXIDE <u>INSTRUMEN</u>T MKS-FTIR/2030/001785245 ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

279.6 25Sep2013

2nd Analysis: 220ct2013

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

280.6 PPM 25Sep2013

Special Notes:

275.00 PPM SO2 (250-300 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN



Quarterly Cylinder Gas Audit Checklist Stack SO₂ Analyzer

02

Revised: 03/27/07

Unit Number (Circle One):

2

(8)

Date: 10 9 14 Time: 10: 24 pm Serial Number: VE-920-8700-2 Technician: Envigue Nieto Signature: Chigsthy

Cylinder ID number	CC152088		CC51515			
Date of Certification	25 OCT. 2013		25 OCT. 2013			
Type of certification	EPA		EPA			
(e.g. EPA Protocol 1					,	
or CRM).	1450	PSI	2000 43	I		
	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value	5,07	15 0	5,07	15.0	5.07	15.0
C_a (ppm)	3,01	15,0	5,01	13.0	3,01	10,0
CEM Response	4.48	111.61	4.51	14.4	4.48	144
value C_m (ppm)	1,570	14.4	7.37	17.7	7,10	17.7
Accuracy A (% or	-11.637	-4.0	-11,045	41 1	-11.637	-40'
ppm)	11.621	1,0	-11,010	-7.0	11.63	1.0

where A =
$$\underbrace{(C_m - C_n)}_{C_n} X 100$$





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926 8832 DICE ROAD SANTA FE SPRINGS, CA 90670-2516

Document #: 52372226-001

Folio #:RDIA018

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012 US.

ANALYTICAL INFORMATION Gas Type: O2,BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1, EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC51515

Certification Date:

25Oct2013

Exp, Date:

260ct2021

Cýlinder Pressure:

2000 PSIG

Batch No:

\$B00079703

COMPONENT OXYGEN NITROGEN

CERTIFIED CONCENTRATION (Moles) 15.0

BALANCE

ACCURACY (ABSOLUTE / RELATIVE)

TRACEABILITY

REFERENCE STANDARD

COMPONENT

CONCENTRATION 20.9500

UNCERTAINTY 0.1300

CYLINDER K016558

TYPE/SRM SAMPLE

NTRM 2659

EXP. DATE 14May2018

ANALYTICAL METHOD

1st Analysis: 250ct2013

COMPONENT OXYGEN

INSTRUMENT

ANALYTICAL/PRINCIPLE CALIBRATED CONCENTRATION

250ct2013

14.97 %

HP/5890F/3336A60154

15% (+/- 5%) OXYGEN, BALANCE NITROGEN EPA PROTOCOL - written certs & tags Rhodia

APPROVED BY:

Special Notes:



Eco Services - HoustonBaton Rouge

Quarterly Cylinder Gas Audit Checklist T.G. OUTLET Stack SO₂ Analyzer

Unit Number (Circle One):

(29)

8

Date: 1/16/15 Time: 2:10 pm Serial Number: 1E-920-10628-1 Technician: E. Nieto D. García. Signature: C.

Cylinder ID number	ALMOS	19933	ALMODO	1367			
Date of Certification	OCT. 24		OCT. 23	1,2021			
Type of certification	EPF	j	EPA	<u>.</u>			
(e.g. EPA Protocol 1 or CRM).	Protoco	11	Protoc	0/1			
	Trial 1		Tria	Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit	
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2	
Certified audit value	1.00	000	127	280	127	280	
C_a (ppm)	127	280	127	0.0			
CEM Response	10/2	282	129	284	131	286	
value C_m (ppm)	126	200	1,0				
Accuracy A (% or ppm)	-0.787	0.714	1.575	1.429	3.150	2.143	

where A = $(\underline{C_m} - \underline{C_a}) \times 100$ $\underline{C_a}$



Evolution . W. L. CLASS

Gut m. con coursey

SANTA F-SPRIN.

Phone: 800-123-2212 Fax 561-164-E262

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FUALT BASES UB32 PICE IN SANT

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.y 201 . Protection Assay & Certification of Gaseous Calibration with as y 201 . Pas standard if pressure is less than 100 mit.

220ct2013 cre is loc Date:

Exp. Date* 240012021 Batch No: SBO****5447

093 . SiG

CERTIFIED CONCENTRATION (Moles) BALALICE

ACCUPACY (ABSOLUTE / I.E. A'LIVE)

2 X 1) P 1 CC 372 S2 N RM C26J

EXP. DATE 20May2016

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Dr. Company

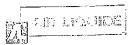
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COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013

AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Decument # : 52372052-004

Fo'!o #:RDIAQRTY003

RHODIA INC. STOREROOM

8615 MANCHESTER HOUSTON TX 77012

HS

ANALYTICAL INFORMATION Gas Type: SO2, BALN

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1, EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number: Cylinder Pressure:

ALM029933 2000 PSIG

Certification Date:

230ct2013

240ct2021

Exp. Date: Batch No:

SB00079449

COMPONENT SULFUR DIOXIDE CERTIFIED CONCENTRATION (Moles) 127

PPM

BALANCE

ACCURACY (ABSOLUTE / RELATIVE) PPM 8.0 1.0

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE

NITROGEN

CONCENTRATION 100.4006

PPM

JN, ERTAINT ' 0.3000

CYLINDER KAL003572

TYPE/SRM SAMPLE

NTRM 1694

EXP. DATE

24Jan2018

ANALYTICAL METHOD

tar Anal, sis

16 (c)29%

COMPONENT SULFUR DIOXIDE INSTAUNCHT

NKS-FTIR/2039/001785245

ANA CYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

128.7 PPM

2nd Analysis 23t 322 3

COMPONENT SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

126.8 PFivl

Special Notes:

125.00 PPM SO2 (100-150 PPM ACCEPTABLE), BALANCE NITROGEN EPA'CEMS GRADE PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN



Eco Services - HoustonBaton Rouge

Quarterly Cylinder Gas Audit Checklist T. G. OUTLET Stack SO₂ Analyzer

Unit Number (Circle One):

(2,)

R

Date: 1/16/15 Time: 3:02pm Serial Number: VE-920-10628-1 Technician: E. Nieto D. Bavcie Signature: Dieto Discourse

Cylinder ID number	ICC24	3658	CC 83	040		
Date of Certification	OCT. 2	3,2021	OCT. 22	2,2021		··
Type of certification	EPA	′				
(e.g. EPA Protocol 1 or CRM).	Protoc	011	Protoco	,11		
	Tr	ial 1	Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	906	2000	906	2000	906	2000
CEM Response value C_m (ppm)	928	1978	937	1980	938	1979
Accuracy A (% or ppm)	2.428	-1,100	3,422	-1.000	3,532	-1.050

where
$$A = (C_m - C_a) \times 100$$

 C_a





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013
AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926 8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document # : 52372052-001 Folio #:RDIAGRTY006

Customer RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012

ANALYTICAL INFORMATION Gas Type: SO2, BALN
This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards;
Procedure G-1. EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC83040

Certification Date:

210ct2013

Exp. Date:

220ct2021 SB00079338 i.

Cylinder Pressure:

2000 PSIG

Batch No:

COMPONENT SULFUR DIOXIDE

CERTIFIED CONCENTRATION (Moles) 2,000

NTRM 1664

NITROGEN

PPM BALANCE

ACCURACY (ABSOLUTE / RELATIVE)
12. PPM / 0.6 %

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION PPM 2402,0000

UNCERTAINTY 14.0000

CYLINDER ALM063514

TYPE/SRM SAMPLE

EXP. DATE 03Nov2017

ANALYTICAL METHOD

1st Analysis: 140ct2013

COMPONENT SULFUR DIOXIDE

COMPONENT

SULFUR DIOXIDE

!NSTRUMENT MKS-FTIR/2030/001785245 <u>ANALYTICAL/PRINCIPLE</u>

CALIBRATED 25Sep2013

CONCENTRATION 1997. PPM

2nd Analysis: 210ct2013

INSTRUMENT

MKS-FTIR/2030/001785245

ANALYTICAL/PRINCIPLE

CALIBRATED 25Sep2013

CONCENTRATION

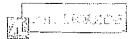
1994. PPM

Special Notes:

1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN 1980.00 PPM SO2 (1800-2100 ACCEPTABLE), BALANCE NITROGEN EPA CEMS PROTOCOL WRITTEN CERT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN





COMPLIANCE CLASS

Guaranteed +/- 2% Accuracy

8832 DICE ROAD, SANTA FE SPRINGS, CA 90670-2516

Phone: 800-323-2212

Fax: 562-464-5262

CERTIFICATE OF ACCURACY: EPA Protocol Gas

Assay Laboratory - PGVP Vendor ID: A52013 AIR LIQUIDE AMERICA SPECIALTY GASES LLC P.O. No.: 4501864926

8832 DICE ROAD

SANTA FE SPRINGS, CA 90670-2516

Document #: 52372052-002

Folio #:RDIAQRTY005

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON TX 77012

Gas Type: SO2, BALN ANALYTICAL INFORMATION

This certification was performed according to EPA Traceability Protocol For Assay & Certification of Gaseous Calibration Standards; Procedure G-1, EPA/600/R-12/531; May 2012. Do not use this standard if pressure is less than 100 psig.

Cylinder Number:

CC243058

Certification Date:

220ct2013

Exp. Date:

230ct2021 SB00079445

Cylinder Pressure:

2000 PSIG

Batch No:

COMPONENT SULFUR DIOXIDE NITROGEN

CERTIFIED CONCENTRATION (Moles)

906

PPM BALANCE

PPM

ACCURACY (ABSOLUTE / RELATIVE) PPM

TRACEABILITY

REFERENCE STANDARD

COMPONENT SULFUR DIOXIDE CONCENTRATION 975.0000 PPM

UNCERTAINTY 7.0000

CYLINDER KAL003179

TYPE/SRM SATIPLE

NTRM 1632

EXP, DATE 01Jun2016

ANALYTICAL METHOD

140ct2013 1st Analysis:

COMPONENT

SULFUR DIOXIDE

INSTRUMENT

MKS-FTIR/2030/001785245

FTIR

A......CAL/PRINCIPLE CALIBRATED -CONCENTRATION

25Sep2013 905.5 PPM

2nd Analysis: 220ct2 13

COMPONENT SULFUR DIOXIDE INSTRUMENT

MKS-FTIR/2030/001785245

ANAL'/TICAL/PRINCIPLE

CALIBRATED

CONCENTRATION

25Sep2013 905.8

Special Notes:

900.00 PPM SO2 (800-1000 PPM ACCEPTABLE), BALANCE NITROGEN EPA CEMS GRADE PROTOCOL WRITTEN CEPT & TAGS TO RHODIA

APPROVED BY:

THUAN TRAN



Quarterly Cylinder Gas Audit Checklist MGB SO₂ Analyzer

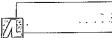
Technician: Marcus/Gonzalez Signature:

Revised: 01/21/15

Cylinder ID number		6449	ALMO			
Date of Certification	O7 0:	+ 2013		4 2013	f	·
Type of certification	EF	³ A	E	. 1	E	
(e.g. EPA Protocol 1	Pro Foc	01 1	Profo	col 1	Proto	co1 1
or CRM).						1.0
	Trial 1		Trial 2		Trial 3	
	Audit	Audit	Audit	Audit	Audit	Audit
	Point 1	Point 2	Point 1	Point 2	Point 1	Point 2
Certified audit value C_a (ppm)	3.75	8.24	3.75	8.24	3.75	3.24
CEM Response	3.70	8.14	3.83	8,34	4.07	8,61
value C_m (ppm)						
Accuracy A (% or ppm)	-1.333	-1.214	2.133	1.214	8,533	4.490

where
$$A = (C_m - C_n) \times 100$$

 C_a





Single-Certified Calibration Standard

1290 COMBERMERE STREET, TROY, MI 48083

Phone: 248-589-2950 Fax: 248-589-2134

Certified Master Class Calibration Standard CERTIFICATE OF ACCURACY:

Product Information
Document #:52384939-002
Item No.: M374501-P-30AL
P.O. No.: 4501864926

Cylinder Number: ALM046889 Cylinder Size: 30AL

Certification Date: 07Oct2013 Expiration Date: 08Oct2016 Lot Number: TRO0094111

Customer

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON, TX 77012

CERTIFIED CONCENTRATION

Component Name

SULFUR DIOXIDÉ. NITROGEN

Concentration (Moles)

Accuracy (+1-%)

3.75

BALANCE

2

TRACEABILITY

Traceable To

Scott Reference Standard

APPROVED BY:

ROBERT LESNIAK





Single-Ceráfied Calibration Standard

1290 COMBERMERE STREET, TROY, MI 48083

Phone: 248-589-2950 Fax: 248-589-2134

CERTIFICATE OF ACCURACY: Certified Master Class Calibration Standard

Product Information
Document #: 52384839-003
Item No.: M374501-P-30AL
P.O. No.: 4501864926

Cylinder Number: CC196449 Cylinder Size: 30AL Certification Date: 07Oct2013 Expiration Date: 08Oct2016 Lot Number: TR00094123 Customer

RHODIA INC. STOREROOM 8615 MANCHESTER HOUSTON, TX 77012 US

CERTIFIED CONCENTRATION

Component Name

SULFUR DIOXIDE NITROGEN Concentration (Moles)

8.24

% BALANCE

(+/-%) 2

Асканасу

TRACEABILITY

Traceable To

Scott Reference Standard

	•		
APPROVED BY:	ROBERT I ESNIAK	DATE:	

44/890 v3 AMAYER





Eco Services Operations LLC Houston Plant

RECEIVE

MAR 2 5 2015

Coomination Branch

CERTIFIED MAIL: RETURN RECEIPT REQUESTED (7011 2000 0001 4575 2313)

March 2, 2015

Mr. Jeff Robinson Air Permits Section Mail Code 6PD-R U.S. EPA – Region VI 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

RE: Benzene Waste Operations NESHAP

Industrial Solid Waste Registration No. 31019

Hazardous Waste Permit No. HW-50095

40 CFR Part 61, Subpart FF EPA ID No. TXD008099079

Dear Mr. Robinson:

Enclosed please find a report for the 2014 calendar year Benzene Waste Operations summary for Eco Services Operations LLC's Houston, Texas facility. Eco Services operates a commercial industrial furnace permitted under 40 CFR Part 264 and Part 266 Subpart H by the State of Texas. This report is required under 40 CFR Part 61, Subpart FF-National Emission Standard for Benzene Waste Operations.

We have reviewed the status of each waste stream subject to regulation under this standard. In accordance with section 61.355(a), the Total Annual Benzene (TAB) quantity from this facility's waste operations was 1.8 megagrams for the operating year 2014.

Quarterly fugitive emission monitoring did not identify any emissions >500 ppm as defined in 40 CFR 61.343(a)(1)(i)(A).

Eco Services documented all daily visual inspections of the hazardous waste operations area as required in the quarterly inspection requirement as defined in 40 CFR 61.343(c). Visual inspections included sight, smell and sound observations and found no leaks in 2014.

If there are any questions, or if further information is required, please contact me at 713-924-1408.

Sincerely,

W. F. Dickerson

Environmental Manager

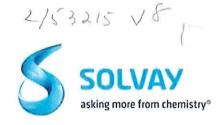
Attachment

Eco Services Operations LLC Houston Plant 8615 Manchester Street Houston, TX 77012 CC: Air Section Manager, TCEQ, Region 12, Houston
Mr. Bob Allen, Director, Environmental Public Health Division,
Harris County Public Health and Environmental Services
Mr. Arturo Blanco, City of Houston, Bureau of Air Control

Eco Services Operations LLC Houston Plant Calendar Year 2014 Annual Benzene Report

40 CFR 61 Subpart FF - Benzene Annual Report

			,						Mg/yr
61.357(a)(3)(vi)	Annual Benzene Quantity (Mg/yr)	0.0	0.0	0.0	1.8	0.0	0.0	0.0	1.8
61.357(a)(3)(v)	Annual Average Flow Weighted Benzene Concentration (ppmw)	10	200	10,000	50,000	10	1,000	2,000	TOTAL
61.357(a)(3)(iv)	Range of Benzene Concentration (ppmw)	0-10	10-200	0-10,000	10,000-50,000	0-10	0-1,000	10-2,000	A
61.357(a)(3)(ii) 61.357(a)(3)(iii) 61.357(a)(3)(iv) 61.357(a)(3)(v) 61.357(a)(3)(v)	Annual Waste Quantity (Mg/yr)	0.0	0.1	3.6	35.7	0.0	 8	0.2	
61.357(a)(3)(ii)	Waste Stream a Process Wastewater Stream, Product Tank Drawdown, or Landfill Leachate	Y	Z	z	z	Z	Z	Y	Y=Yes, N=No
61.357(a)(3)(i)	Water Content of Waste Stream >10%	λ	z	z	z	Z	Z	У	
61.357(a)(2)	Controlled Benzene Emissions	X	>	>	≻	>	>	Υ	Y=Yes, N=No Y=Yes, N=No
61.35	Waste Stream	9109003	9104004	0706008	0312002	1205001	0912006	9405021	



Al/Al/co

6EN-AA Prodice TOTONA

Solvay USA Inc. Houston Plant

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 0517)

RECEIVE

January 27, 2014

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733 11 21 20:4

Air/Toxics & Inspection Coordination Branch 6EN-A

Re:

Solvay Benzene NESHAP, Subpart FF, Quarterly Report

October 1, 2013 to December 31, 2013

EPA ID No.: TXD008099079

Dear Mr. Robinson:

Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Solvay receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Solvay submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Solvay USA Inc. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012 Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

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Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

45 3215 V8 (AIIAI/CO [110000460901

TX 079 Vol. 8

ECOSERVICES

RECEIVE

Eco Services Operations LLC Houston Plant

FEB - 5 2015

Certified Mail Return Receipt Requested (7011 2970 0000 3516 7784)

Any foxics & inspection Coordination Branc' BEN-A

January 29, 2014

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Subject:

Eco Services

Houston, Texas Plant

NSPS Kb Semiannual Report - 2nd Half 2014

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS)	liquids (VOL)	Vapor Combustor (TSVC) as backup
	Tanks		
Tk 48	Spent Acid	Spent sulfuric	Regeneration Unit No.2 Furnace with Spent
Tk 49	(SA)Tanks	acid with	Acid Vapor Combustor as backup.
Tk 53	2001	potential for	
Tk 56*		containing	
Tk 78*		volatile organic	
		liquids	

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.



Eco Services Operations LLC Houston Plant **Spent Acid Tanks Summary Report**

Pollutant	VOC
Reporting period dates:	7/1/2014 to 12/31/2014
Company:	Eco Services LLC Houston site
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Spent Acid Tank Farm
Total source operating time in reporting period:	4,344 hours
Duration of excess emissions in reporting period due to:	
a. Startup/shutdown	0.0 hours
b. Control equipment problems	0.0 hours
c. Process problems	0.0 hours
d. Other known causes	0.0 hours
e. Unknown causes	0.0 hours
Total duration of excess emission	0.0 hours
Total duration of excess emissions	0.0 %



Eco Services Operations LLC Houston Plant TS Tanks Summary Report

Pollutant	VOC		
Reporting period dates:	7/1/2014 to 12/31/2014		
Company:	Eco Services LLC Houston site		
Emission Limitation:	22.22 lbs/hr when venting to TSVC		
Address:	8615 Manchester Houston, TX 77012		
Monitor Manufacturer and Model No:	Not Applicable		
Date of Latest CMS Certification or Audit:	Not Applicable		
Process Unit Description:	Treatment Services Tank Farm		
Total source operating time in reporting period:	4,344 hours		
Duration of excess emissions in reporting period due to:	3		
f. Startup/shutdown	0 hours		
g. Control equipment problems	0.0 hours		
h. Process problems	0.0 hours		
i. Other known causes	0.25 hours		
j. Unknown causes	0 hours		
Total duration of excess emission	0.25 hours		
Total duration of excess emissions	0.006 %		

If you have any questions concerning this matter, please call David Laurie at (713) 924-1484.

Sincerely,

William McConnell

William McConnell

Plant Manager

453215 18



Solvay USA Inc. Houston Plant 110000460401 6EPEFFE JUL 14 2014

> Air/Toxics & Inspection Coordination Branc 6EN-A

Certified Mail; Return Receipt Requested (7011 2000 0001 4575 1392)

A1/4/C6

July 8, 2014

Mr. Jeff Robinson Chief, Air Permits Section 6PD-R U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

HON Semiannual Report per 40 CFR 63.152(c)

TCEQ Identification Nos.: RN100220581/CN604514315

Dear Mr. Robinson:

Solvay USA Inc. (Solvay) is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Solvay submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Solvay submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of January 1 to June 30, 2014.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

HON Group 1 wastewater and residuals may be stored in one or more of the six (6) Treatment Services (TS) tanks. There were no control equipment failures identified for these tanks in the reporting period.

63.146(d) – Treatment process monitoring data.

The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

Solvay USA Inc. Houston Plant 8615 Manchester Street Houston, TX 77012 63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

The TS tanks normally vent to the Regeneration Unit No. 2 (Regen 2) sulfuric acid furnace which is exempt from monitoring per 63.139(d)(4)(iv). In the event that Regen 2 is unavailable, tank vapors are routed to the Treatment Services Vapor Combustor (TSVC). The TSVC minimum combustion temperature is 1,500°F per operating permit. During this reporting period, there were no instances of the daily average TSVC combustion temperature being under 1,500°F while TS tanks were venting to it. There were no days when insufficient monitoring data were collected during this reporting period.

63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) – For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

63.146(e)(1), Table 20 item (8)(ii) and 63.148(j)(3) - Report the times and durations of any periods when the bypass valves are moved to the diverting position, the seal has been changed, the seal mechanism is broken, or the key to unlock the bypass line valve was checked out.

There were no bypass valve or car-seal abnormalities this reporting period. We do not use lock-and-key mechanisms for this purpose.

Please contact Floyd Dickerson at 713-924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

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Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

45-3215 V8 AI/AI/CO



Rhodia

Certified Mail Return Receipt Requested (7011 2000 0001 4575 1606)

July 30, 2014

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Subject:

Solvay USA Inc.

Houston, Texas Plant

NSPS Kb Semiannual Report – 1st Half 2013

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS)	liquids (VOL)	Vapor Combustor (TSVC) as backup
	Tanks	7569 387 5507	
Tk 48	Spent Acid	Spent sulfuric	Regeneration Unit No.2 Furnace with Spent
Tk 49	(SA)Tanks	acid with	Acid Vapor Combustor as backup.
Tk 53		potential for	
Tk 56*		containing	
Tk 78*		volatile organic	
		liquids	

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.

Solvay Inc. **Houston Plant** 8615 Manchester Street Houston, TX 77012

Solvay USA Inc. **Houston Plant** RECEIVE

AUG - 4.2014

Air oxics & Inspection Coordination Branc' 6EN-A

Spent Acid Tanks Summary Report

Pollutant	VOC
Reporting period dates:	1/1/2014 to 6/30/2014
Company:	Solvay USA Inc. Houston site
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Spent Acid Tank Farm
Total source operating time in reporting period:	4,344 hours
Duration of excess emissions in reporting period due to:	
a. Startup/shutdown	0.0 hours
b. Control equipment problems	0.67 hours
c. Process problems	6.0 hours
d. Other known causes	0.0 hours
e. Unknown causes	0.0 hours
Total duration of excess emission	6.67 hours
Total duration of excess emissions	0.15 %

TS Tanks Summary Report

Pollutant	VOC
Reporting period dates:	1/1/2014 to 6/30/2014
Company:	Solvay USA Inc. Houston site
Emission Limitation:	22.22 lbs/hr when venting to TSVC
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Treatment Services Tank Farm
Total source operating time in reporting period:	4,344 hours
Duration of excess emissions in reporting period due to:	
f. Startup/shutdown	0 hours
g. Control equipment problems	1.75 hours
h. Process problems	0.0 hours
i. Other known causes	0.0 hours
j. Unknown causes	0 hours
Total duration of excess emission	1.75 hours
Total duration of excess emissions	0.04 %

If you have any questions concerning this matter, please call David Laurie at (713) 924-1484.

Sincerely,

Millian J. m- Conlil William McConnell Plant Manager

Solvay USA Inc.

Attachment

Solvay Inc. Houston Plant 8615 Manchester Street Houston, TX 77012

Cc:

Executive Director, MC-109
Texas Commission on Environmental Quality
P.O. Box 13087
Austin, TX 78711-3087

Air Section Manager Texas Commission on Environmental Quality 5425 Polk Avenue, Suite H Houston, TX 77023-1486

Bureau Chief Bureau of Air Quality Control City of Houston 7411 Park Place Blvd. Houston, TX 77087-4441

Director Harris County Public Health and Environmental Services Environmental Public Health Division 101 S. Richey Suite G Pasadena, TX 77506

> Solvay Inc. Houston Plant 8615 Manchester Street Houston, TX 77012

A1/41/co

1.8

RECEIVE N-AA

APR 29 2015

Ant Toxics & Inspection Coordination Branch

6EN-A

ECOSERVICE Eco Services Operations LLC

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 2375)

April 23, 2015

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

January 1, 2015 to March 31, 2015 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations LLC, formally Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely, Willin J. M-Cornell

William McConnell Plant Manager

ce: Air Section Manager, TCEQ, Region 12

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

Table 1

Eco Services Operations LLC Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: March 31, 2015

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	•
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	Wallians and the same of the s
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

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110000460901 GEN-AA TX079V,8

ECOSERVICES

Eco Services Operations LLC Houston Plant

Certified Mail; Return Receipt Requested (7011 2970 0000 3521 0596)

JAN 16 2015

Am Foxics & Inspection Coordination Branc 6EN-A

January 12, 2015

Mr. Jeff Robinson
Chief, Air Permits Section
6PD-R

U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200

Dallas, Texas 75202-2733

Re:

HON Semiannual Report per 40 CFR 63.152(c)

TCEO Identification Nos.: RN100220581/CN604683482

Dear Mr. Robinson:

Eco Services Operations LLC (Eco Services), formally Solvay USA Inc., is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Eco Services submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Eco Services submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of July 1 to December 31, 2014.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

HON Group 1 wastewater and residuals may be stored in one or more of the six (6) Treatment Services (TS) tanks. There were no control equipment failures identified for these tanks in the reporting period.

63.146(d) – Treatment process monitoring data.

The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

The TS tanks normally vent to the Regeneration Unit No. 2 (Regen 2) sulfuric acid furnace which is exempt from monitoring per 63.139(d)(4)(iv). In the event that Regen 2 is unavailable, tank vapors are routed to the Treatment Services Vapor Combustor (TSVC). The TSVC minimum combustion temperature is 1,500°F per operating permit. During this reporting period, there were no instances of the daily average TSVC combustion temperature being under 1,500°F while TS tanks were venting to it. There were no days when insufficient monitoring data were collected during this reporting period.

63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) – For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

63.146(e)(1), Table 20 item (8)(ii) and 63.148(j)(3) - Report the times and durations of any periods when the bypass valves are moved to the diverting position, the seal has been changed, the seal mechanism is broken, or the key to unlock the bypass line valve was checked out.

There were no bypass valve or car-seal abnormalities this reporting period. We do not use lock-and-key mechanisms for this purpose.

Please contact Floyd Dickerson at 713-924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Mr. Arturo Blanco, Bureau of Air Quality Control, City of Houston

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Coordination Bran

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Certified Mail; Return Receipt Requested (7011 2000 0001 4575 2030) AUG 6 2015

July 30, 2015

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section 6PD-R U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Rei

HON Semiannual Report per 40 CFR 63.152(c)

TCEQ Identification Nos.: RN100220581/CN604683482

Dear Mr. Robinson:

Eco Services Operations LLC (Eco Services), formally Solvay USA Inc., is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Eco Services submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Eco Services submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of January 1 to June 30, 2015.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

HON Group 1 wastewater and residuals may be stored in one or more of the six (6) Treatment Services (TS) tanks. There were no control equipment failures identified for these tanks in the reporting period.

63.146(d) – Treatment process monitoring data.

The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

The TS tanks normally vent to the Regeneration Unit No. 2 (Regen 2) sulfuric acid furnace which is exempt from monitoring per 63.139(d)(4)(iv). In the event that Regen 2 is unavailable, tank vapors are routed to the Treatment Services Vapor Combustor (TSVC). The TSVC minimum combustion temperature is 1,500°F per operating permit. During this reporting period, there were no instances of the daily average TSVC combustion temperature being under 1,500°F while TS tanks were venting to it. There were no days when insufficient monitoring data were collected during this reporting period.

63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) — For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

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There were no bypass valve or car-seal abnormalities this reporting period. We do not use lock-and-key mechanisms for this purpose.

Please contact Floyd Dickerson at 713-924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

Milling in Grould

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Director, Health and Human Services Department, City of Houston



Certified Mail Return Receipt Requested (7015 1520 0003 4945 7531)

RECEIVE

July 30, 2015

AUG 3 2015

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Am Toxics & inspection Coordination Branc 6EN-A

Subject:

Eco Services LLC

Houston, Texas Plant

NSPS Kb Semiannual Report – 1st Half 2015

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS)	liquids (VOL)	Vapor Combustor (TSVC) as backup
	Tanks		, , , , , ,
Tk 48	Spent Acid	Spent sulfuric	Regeneration Unit No.2 Furnace with Spent
Tk 49	(SA)Tanks	acid with	Acid Vapor Combustor as backup.
Tk 53		potential for	
Tk 56*		containing	
Tk 78*		volatile organic	×
		liquids	ě.

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.



RECEIVE

TS Tanks Summary Report

Pollutant	VOC	AUG. 3. 2015
Reporting period dates:	1/1/2015 to 6/30/2015	10
Company:	Eco Services LLC Houston site	Coordination Branc
Emission Limitation:	22.22 lbs/hr when venting to TSVC	6EN-A
Address:	8615 Manchester Houston, TX 77012	
Monitor Manufacturer and Model No:	Not Applicable	
Date of Latest CMS Certification or Audit:	Not Applicable	
Process Unit Description:	Treatment Services Tank Farm	
Total source operating time in reporting period:	4,344 hours	
Duration of excess emissions in reporting period due to:		
f. Startup/shutdown	0 hours	
g. Control equipment problems	0 hours	
h. Process problems	0.0 hours	
i. Other known causes	0.0 hours	
j. Unknown causes	0 hours	
Total duration of excess emission	0.0 hours	
Total duration of excess emissions	0.0 %	

If you have any questions concerning this matter, please call David Laurie at (713) 924-1484.

Sincerely, Milliam J. M. Comill

William McConnell

Plant Manager

Solvay USA Inc.

Attachment



RECEIVE

Spent Acid Tanks Summary Report

Pollutant	VOC AUG]. 3 2015
Reporting period dates:	1/1/2015 to 6/30/2015 -uii 10xic	s & Inspection
Company:		ation Branc
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)	EN-A
Address:	8615 Manchester Houston, TX 77012	
Monitor Manufacturer and Model No:	Not Applicable	
Date of Latest CMS Certification or Audit:	Not Applicable	
Process Unit Description:	Spent Acid Tank Farm	
Total source operating time in reporting period:	4,344 hours	
Duration of excess emissions in reporting period due to:		
a. Startup/shutdown	0.0 hours	
b. Control equipment problems	0.00 hours	
c. Process problems	322.75 hours	
d. Other known causes	0.0 hours	
e. Unknown causes	0.0 hours	
Total duration of excess emission	322.75 hours	77
Total duration of excess emissions	7.5 %	



Cc:

Executive Director, MC-109 Texas Commission on Environmental Quality P.O. Box 13087 Austin, TX 78711-3087

Air Section Manager Texas Commission on Environmental Quality 5425 Polk Avenue, Suite H Houston, TX 77023-1486

Bureau Chief Bureau of Air Quality Control City of Houston 7411 Park Place Blvd. Houston, TX 77087-4441

Director
Harris County Public Health and Environmental Services
Environmental Public Health Division
101 S. Richey Suite G
Pasadena, TX 77506

RECEIVE

AUG 3 2015

Am Toxics & Inspection Coordination Bran-6EN-A TX:07910/8 4/53 215 V8

ASSOCIATE DIRECTOR AI AI/CO

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COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

CERTIFIED MAIL: Return Receipt Requested (7015 1520 0003 4945 7661)

October 29, 2015

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Eco Services Operations LLC

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

July 1, 2015 to September 30, 2015 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations LLC, formally Solvay USA Inc., in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

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Plant Manager

cc: Air Section Manager, TCEQ, Region 12

willen J. br Comell

Director, Health and Human Services Department, City of Houston

Table 1

Eco Services Operations LLC Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: September 30, 2015

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	THE STATE OF THE S
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		** ** **
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor	Strate in	
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

ECOSERVICES Operations LLC

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US EPA, DALLAS, TX
ASSOCIATE DIRECTOR
COMPLIANCE ASSURANCE
COMPLIANCE ASSURANCE
ENERGEMENT DIV.

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CERTIFIED MAIL: RETURN RECEIPT REQUESTED (7015 1520 0003 4945 80025)

February 29, 2016

Houston Plant

Mr. Jeffrey Robinson Air Permits Section Mail Code 6PD-R U.S. EPA – Region VI 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

RE:

Benzene Waste Operations NESHAP Industrial Solid Waste Registration No. 31019 Hazardous Waste Permit No. HW-50095 40 CFR Part 61, Subpart FF EPA ID No. TXD008099079

Dear Mr. Robinson:

Enclosed please find a report for the 2015 calendar year Benzene Waste Operations summary for Eco Services Operations DLC's-Houston, Texas facility. Eco Services operates a commercial industrial furnace permitted under 40 CFR Part 264 and Part 266 Subpart H by the State of Texas. This report is required under 40 CFR Part 61, Subpart FF-National Emission Standard for Benzene Waste Operations.

We have reviewed the status of each waste stream subject to regulation under this standard. In accordance with section 61.355(a), the Total Annual Benzene (TAB) quantity from this facility's waste operations was 0.5 megagrams for the operating year 2015.

Quarterly fugitive emission monitoring did not identify any emissions >500 ppm as defined in 40 CFR 61.343(a)(1)(i)(A).

Eco Services documented all daily visual inspections of the hazardous waste operations area as required in the quarterly inspection requirement as defined in 40 CFR 61.343(c). Visual inspections included sight, smell and sound observations and found no leaks in 2015.

If there are any questions, or if further information is required, please contact me at 713-924-1408.

Sincerely,

W. F. Dickerson

Environmental Manager

Attachment .

CC: Air Section Manager, TCEQ, Region 12, Houston
Mr. Bob Allen, Director, Environmental Public Health Division,
Harris County Public Health and Environmental Services
City of Houston, Bureau of Air Control

Eco Services Operations LLC Houston, Texas Calendar Year 2015 Annual Benzene Report

40 CFR 61 Subpart FF - Benzene Annual Report

											Mg/yr
61.357(a)(3)(vi)	Annual Benzene Quantity (Mg/yr)	0.0	0.0	0.0	0.0	0.0	0.5	0.0	0.0	0.0	0.5
61.357(a)(3)(v)	Annual Average Flow Weighted Benzene Concentration (ppmw)	10	200	10,000	200	1,000	20,000	10	1,000	2,000	TOTAL
61.357(a)(3)(iv)	Range of Benzene Concentration (pprnw)	0-10	10-200	0-10,000	0-200	0-1,000	10,000-50,000	0-10	0-1,000	10-2,000	
61.357(a)(3)(iii)	Annual Waste Quantity (Mg/yr)	0.0	0.1	0.1	0.0	0.0	10.1	0.0	1.5	0.3	
61.357(a)(3)(ii)	Waste Stream a Process Wastewater Stream, Product Tank Drawdown, or Landfill Leachate	X	z	z	z	z	z	z	Z	\	Y=Yes, N=No
[61.357(a)(3)(i)	Water Content of Waste Stream >10%	\	z	z	Z	Z	z	z	z	\	Y=Yes, N=No
61.357(a)(2)	Controlled Benzene Emissions	>	>-	>-	>	>	>	>	>	\	Y=Yes, N=No Y=Yes, N=No
61.35	Waste Stream	9109003	9104004	0706008	1503002	1409001	0312002	1205001	0912006	9405021	

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COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

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CERTIFIED MAIL: Return Receipt Requested (7015 1520 0003 4945 7890)

January 28, 2016

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FP, Quarterly Report

July 1, 2015 to September 30, 2015 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations LLC in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
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Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Director, Health and Human Services Department, City of Houston

Table 1

Eco Services Operations LLC Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: <u>December 31, 2015</u>

Inspection	Was inspection performed?	Exceptions Noted		
Annual Method 21 inspections of tank covers	x Yes			
and openings per 61.343(a)(1)(i)	Except as Noted			
Quarterly visual inspections of tank covers	x Yes			
and openings per 61.343(c)	Except as Noted			
Initial and annual Method 21 inspections of	x Yes			
containers per 61.345(a)(1)	Except as Noted			
Initial and quarterly visual inspections of	x Yes	1		
containers per 61.345(b)	Except as Noted			
Annual Method 21 inspections of treatment	x Yes			
system openings (Regeneration Unit No. 2)	Except as Noted			
per 61.348(e)(3)(ii)				
Annual Method 21 inspections of closed vent	x Yes			
systems (from tanks to TS vapor combustor	Except as Noted			
and Regeneration Unit No. 2 industrial				
furnace) per 61.349(a)(1)(i)				
Quarterly visual inspections of closed vent	x Yes			
systems and control devices (from tanks to TS	Except as Noted			
vapor combustor and Regeneration Unit No. 2				
industrial furnace, including the vapor		1 1		
combustor and Regeneration Unit No. 2				
industrial furnace) per 61.349(f)				
Daily inspections of control device continuous	x Yes	-		
monitoring data (temperature of TS vapor	Except as Noted			
combustor and "selected parameter" on				
Regeneration Unit No. 2 industrial furnace)				
per 61.354(c)				

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

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US EPA, DALLAS, TX
ASSOCIATE DIRECTOR

16 FEB - 4 PM 6: 29

COMPLIANCE ASSURANCE

& ENFORCEMENT DIV.

Certified Mail; Return Receipt Requested (7015 1520 0003 4945 7883)

January 28, 2016

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section 6PD-R U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re: (

HON Semiannual Report per 40 CFR 63.152(c)

TCEQ Identification Nos.: RN100220581/CN604683482

Dear Mr. Robinson:

Eco Services Operations LLC (Eco Services) is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Eco Services submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Eco Services submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of July 1 to December 31, 2015.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

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The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

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63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) – For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

63.146(e)(1), Table 20 item (8)(ii) and 63.148(j)(3) - Report the times and durations of any periods when the bypass valves are moved to the diverting position, the seal has been changed, the seal mechanism is broken, or the key to unlock the bypass line valve was checked out.

There were no bypass valve or car-seal abnormalities this reporting period. We do not use lock-and-key mechanisms for this purpose.

Please contact Floyd Dickerson at 713-924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Director, Health and Human Services Department, City of Houston

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Eco Services LLC Houston Plant

Certified Mail Return Receipt Requested (7015 1520 0003 4945 7982)

January 29, 2016

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Subject:

Eco Services LLC

Houston, Texas Plant

NSPS Kb Semiannual Report – 2nd Half 2015

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS)	liquids (VOL)	Vapor Combustor (TSVC) as backup
	Tanks		
Tk 48	Spent Acid	Spent sulfuric	Regeneration Unit No.2 Furnace with Spent
Tk 49	(SA)Tanks	acid with	Acid Vapor Combustor as backup.
Tk 53		potential for	
Tk 56*	**	containing	
Tk 78*		volatile organic	
		liquids	3

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.



Spent Acid Tanks Summary Report

Pollutant	VOC
Reporting period dates:	7/1/2015 to 12/31/2015
Company:	Eco Services LLC Houston site
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Spent Acid Tank Farm
Total source operating time in reporting period:	4,416 hours
Duration of excess emissions in reporting period due to:	
a. Startup/shutdown	0.0 hours
b. Control equipment problems	0.00 hours
c. Process problems	213.89 hours
d. Other known causes	0.0 hours
e. Unknown causes	0.0 hours
Total duration of excess emission	213.89 hours
Total duration of excess emissions	4.8 %



TS Tanks Summary Report

Pollutant	VOC
Reporting period dates:	7/1/2015 to 12/31/2015
Company:	Eco Services LLC Houston site
Emission Limitation:	22.22 lbs/hr when venting to TSVC
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Treatment Services Tank Farm
Total source operating time in reporting period:	4,416 hours
Duration of excess emissions in reporting period due to:	
f. Startup/shutdown	0 hours
g. Control equipment problems	0 hours
h. Process problems	0.0 hours
i. Other known causes	0.0 hours
j. Unknown causes	0 hours
Total duration of excess emission	0.0 hours
Total duration of excess emissions	0.0 %

If you have any questions concerning this matter, please call David Laurie at (713) 924-1484.

Sincerely,

William McConnell

pulling In- Comile

Plant Manager Solvay USA Inc.

Attachment

Eco Services LLC Houston Plant 8615 Manchester Street Houston, TX 77012 CERTIFIED MAIL: Return Receipt Requested (7015 3010 0000 3182 3783)

COMPLIANCE ASSURANCE & ENFORCEMENT DIV

July 14, 2016

Mr. John Blevins Compliance Assurance and Enforcement Section Air/Toxics and Inspection Coordination Branch US EPA Region 6 1445 Ross Avenue (6EN-AT) Dallas, TX 75202-2733

Re:

Eco Services Operations Corp.

Houston, Texas Facility

Notification of Tanks Subject to NSPS Subpart Kb (40 CFR 60.110b)

Dear Mr. Blevins:

Eco Services Operations Corp., formally Eco Services Operations LLC, currently operates Sulfuric Acid Regeneration Unit No. 2 (Regen 2) (TCEQ Air Permit No. 4802) at its Houston facility. Regen 2 receives spent sulfuric acid via truck, barge and rail for recycling in its industrial furnace. Regen 2 is also permitted as a RCRA Boiler and Industrial Furnace (TCEQ Permit No. HW-50095-001) to manage liquid hazardous waste materials. The spent sulfuric acids and liquid hazardous wastes can either be direct-burned or placed in storage tanks prior to being burned. Spent sulfuric acid often contains volatile organic liquids (VOL). The liquid hazardous wastes routinely contain VOL.

Eco Services has revised the operating plan to update the change in ownership of the Houston plant. Eco Services is submitting this operating plan for agency approval. This operating plan covers all onsite tanks subject to NSPS Subpart Kb.

If you should have any questions, please contact David Laurie at (713) 924-1484.

Sincerely,

W. F. Dickerson

Environmental Manager

Attachment

Mr. Richard A. Hyde, P.E. Executive Director, MC-109 Texas Commission on Environmental Quality P.O. Box 13087 Austin, TX 78711-3087

 $C_{\mathbf{C}}$:

Air Section Manager Texas Commission on Environmental Quality Region 12 5425 Polk Avenue, Suite H Houston, TX 77023-1486

> Eco Services Operations Corp. Houston Plant 8615 Manchester Street Houston, TX 77012

NSPS Subpart Kb Operating Plan per 40 CFR 60.113b(c)(1) Eco Services Operations Corp. Houston, Texas Plant Revision 9

Eco Services is modifying the Operating Plan to comply with the NSPS Subpart Kb requirements as they apply to hazardous waste and spent sulfuric acid storage tanks at the Houston Plant. Submittal of an Operating Plan is required by 40 CFR 60.113b(c)(1).

The Houston plant has the following tanks that are subject to vapor control per 40 CFR 60.112b. These tanks use a closed vent system and control devices as the method of compliance per 40 CFR 60.112b(3).

Tank	Unit	Contents	Control Device
B1	Treatment Services	VOL	Regen 2 Furnace with a Vapor
,		Annual Control of the	Combustor as Backup
B2	Treatment Services	VOL	Regen 2 Furnace with a Vapor
			Combustor as Backup

In addition, the following tanks have the potential to contain material that is subject to vapor control per 40 CFR 60.112b. These tanks use a closed vent system and control device as the method of compliance per 40 CFR 60.112b(3).

Tank	Unit	Contents	Control Device	
48	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor	
	Manufacturing		Combustor as Backup	
49	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor	
	Manufacturing		Combustor as Backup	
53	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor	
	Manufacturing		Combustor as Backup	
56*	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor	
	Manufacturing		Combustor as Backup	
78*	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor	
	Manufacturing		Combustor as Backup	

^{*} These tanks have not been reconstructed or modified since 1984, but are listed for completeness.

This plan must document that the tank vent control system will achieve the required 95% control efficiency.

NSPS Subpart Kb Operating Plan per 40 CFR 60.113b(c)(1) Eco Services Operations Corp. Houston, Texas Plant Revision 9

Eco Services is modifying the Operating Plan to comply with the NSPS Subpart Kb requirements as they apply to hazardous waste and spent sulfuric acid storage tanks at the Houston Plant. Submittal of an Operating Plan is required by 40 CFR 60.113b(c)(1).

The Houston plant has the following tanks that are subject to vapor control per 40 CFR 60.112b. These tanks use a closed vent system and control devices as the method of compliance per 40 CFR 60.112b(3).

Tank	Unit	Contents	Control Device
B1	Treatment Services	VOL	Regen 2 Furnace with a Vapor
			Combustor as Backup
В2	Treatment Services	VOL	Regen 2 Furnace with a Vapor
			Combustor as Backup

In addition, the following tanks have the potential to contain material that is subject to vapor control per 40 CFR 60.112b. These tanks use a closed vent system and control device as the method of compliance per 40 CFR 60.112b(3).

Tank	Unit	Contents	Control Device
48	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor
	Manufacturing		Combustor as Backup
49	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor
	Manufacturing		Combustor as Backup
53	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor
	Manufacturing		Combustor as Backup
56*	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor
	Manufacturing		Combustor as Backup
78*	Sulfuric Acid	VOL	Regen 2 Furnace with a Vapor
	Manufacturing		Combustor as Backup

^{*} These tanks have not been reconstructed or modified since 1984, but are listed for completeness.

This plan must document that the tank vent control system will achieve the required 95% control efficiency.

Tanks B1 and B2

Tanks B1 and B2 are fixed roof tanks vented through a closed vent manifold system to either the Regeneration Unit No. 2 (Regen 2) industrial furnace or to the Treatment Services vapor combustor (TSVC). Regen 2 is the primary control system and the TSVC serves as an emergency backup control device. Both of these control systems achieve VOC control efficiencies in excess of 95%, as described below.

(1) Regeneration Unit No. 2 (EPN 104)

The Regen 2 industrial furnace provides temperatures in excess of 816°C and destruction of VOCs in excess of 99.99% as demonstrated in the trial burn for the Regen 2 RCRA Boiler and Industrial Furnace (BIF) permit. Section 60.113b(c)(i) of Subpart Kb allows the documentation of the existence of these conditions to be sufficient for the control system to meet the VOC destruction requirement. Attachment 1 contains a summary table of the trial burn results for Regen 2.

Eco Services monitors temperatures in the Regen 2 industrial furnace and will ensure that the average hourly furnace temperatures remains above 815°C (1,500°F) whenever Tanks B1 and B2 are vented to Regen 2. The temperature in the Regen 2 industrial furnace is maintained between 1,800°F to 2,100°F. The design of the Regen 2 industrial furnace is such that residence times in excess of 0.75 seconds are always maintained. The residence time in the Regen 2 industrial furnace is 2 to 4 seconds.

- (2) Treatment Services Vapor Combustor (EPN 120)
 - The residence time for the Treatment Services vapor combustor is as follows:

Stack Diameter (inner diameter) = 6 feet Combustion Zone = 28 feet

Residence Time =
$$\frac{\text{Stack Volume}}{\text{Gas Flow (acfm)}}$$
 = $\frac{\text{Pi (3)}^2(28)}{25,450}$

= 0.0311 minutes = 1.86 seconds

• The minimum temperature for the Treatment Services vapor combustor is as follows:

Eco Sevices utilizes a log sheet when the vapor combustor is in service when the Regen 2 industrial furnace is not operational. A copy can be found in Attachment 2. The temperature is tracked on the Distributed Control System and all data is maintained for five (5) years.

Tanks 48, 49, 53, 56, and 78

Tanks 48, 49, 53, 56, and 78 are fixed roof tanks vented through a closed vent manifold system to either the Regen 2 industrial furnace or to the spent acid vapor combustor (SAVC). Regen 2 is the primary control system and the SAVC serves as an emergency backup control device. Both of these control systems achieve VOC control efficiencies in excess of 95%, as described below.

(1) Regeneration Unit No. 2 (EPN 104)

The Regen 2 industrial furnace is the primary control for the spent acid tank farm vents and provides temperatures in excess of 816°C and destruction of VOCs in excess of 99.99% as demonstrated in the trial burn for the Regen 2 RCRA Boiler and Industrial Furnace (BIF) permit. Section 60.113b(c)(i) of Subpart Kb allows the documentation of the existence of these conditions to be sufficient for the control system to meet the VOC destruction requirement. Trial burn results for Regen 2 can be found in Attachment 1.

Eco Services monitors temperatures in the Regen 2 industrial furnace and will ensure that the average hourly furnace temperatures remains above 815°C (1,500°F) whenever Tanks 48, 49, 56 and 78 are vented to Regen 2. The temperature in the Regen 2 industrial furnace is maintained between 1,800°F to 2,100°F. The design of the Regen 2 industrial furnace is such that residence times in excess of 0.75 seconds are always maintained. The residence time in the Regen 2 industrial furnace is 2 to 4 seconds.

(2) Spent Acid Vapor Combustor (EPN 170)

The Spent Acid Vapor Combustor (SAVC) operates in series after a caustic scrubber that removes SO_2 from the tank vent system. The caustic scrubber and SAVC are the backup control scheme for the spent acid tank farm vent. When Regen 2 furnace is down, the tank vent system is diverted to the scrubber/combustor. The vapor combustor was designed by the manufacturer to achieve as least 95% DRE at 815° C (1,500°F).

A compliance test was conducted at maximum organic loading to the SAVC. The results from this test demonstrate compliance with 60.113b(c)(1)(i) requirements of 95% destruction of volatile organic compounds when the destruction demonstrated exceeded 95%. A copy of the compliance test report can be found in Attachment 3.

Based on the test results and design, the SAVC achieves ≥95% DRE at 1522°F. Following the precedent in 40 CFR Part 63 Subpart G (HON), compliance will be determined on a daily average basis. Firebox temperature in the SAVC will be continuously monitored. If the daily average firebox temperature (including only temperature data obtained when the valve to the SAVC is open) is at least 1522°F, then compliance is achieved. If this daily average temperature is less than 1522°F, we will calculate the daily average DRE for the SAVC and Regen 2 furnace in aggregate (a time-weighted average). Compliance is achieved if the daily average DRE is at least 95%.

Thus, we have two options to demonstrate compliance:

- 1. SAVC daily average temperature (including only temperature data obtained when the valve to the vapor combustor is open) greater than or equal to 1522°F.
- 2. Calculated daily average DRE (considering SAVC and Regen furnace in aggregate) greater than or equal to 95%.

Two options for compliance are specified because option 2 requires a manual calculation to be performed. We prefer to avoid this manual calculation where possible. Option 1 provides a "screening" indication of compliance that can be calculated automatically by the process control computer. If compliance is demonstrated via option 1, it is not necessary to perform the calculation in option 2.

The following theoretical example shows how compliance could be demonstrated using option 2 if option 1 was unable to demonstrate compliance:

```
12:00 am to 11:00 pm - routed to Regen 2 furnace
11:00 pm to 11:30 pm - routed to SAVC, temperature between 1424-1512°F *11:30 pm to 12:00 am - routed to SAVC, temperature between 1522-1536°F
```

daily average temperature of vapor combustor = 1501°F (as calculated by process control computer)

daily average DRE =

 $\frac{(1380 \text{ min})*(99.9999) + (30 \text{ min})*(90.8) + (30 \text{ min})*(99.6)}{1440 \text{ minutes}} = 99.8\%$

^{*} Destruction and Removal Efficiency conducted on Eco Services Baton Rouge, Louisiana facility's similar vapor combustor installation. Test results can be found in Attachment 4

Attachment 1

RCRA Trial Burn Test Results Demonstrating Regeneration Unit No. 2 DRE

RCRA TRIAL BURN REPORT RHODIA INC. SULFURIC ACID REGENERATION UNIT NO. 2 HOUSTON, TEXAS

TCEQ SOLID WASTE IDENTIFICATION NO.: 31019
TCEQ PERMIT NO.: HW-50095-001
U.S.E.P.A. ID NO.: TXD008099079

Prepared for:

RHODIA INC. 8615 Manchester Street Houston, Texas 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way P.O. Box 2653 West Chester, Pennsylvania 19380

January 2011

W. O. No. 12143.075.003

Table 2-4

Summary of Emissions Test Results – Mode B

Parameter		Test I	Test Results		RCRA Permit
	Run 1	Run 2	Run 3	Average	Limit
Particulate Matter (PM)	0.00093	86000.0	0.00079	0.00000	0.08 gr/dscf @ 7% O2
Hydrogen Chloride (HCl)	0.013	0,014	0.024	0.017	0.103 g/sec
Chlorine (Cl ₂)	< 0.0014	< 0.0017	< 0.0013	< 0.0015	0.093 g/sec
Carbon Monoxide (CO)	29.4	26.3	31.4	29.0	100 ppm @ 7% O ₂ (1 hour rolling average)
Destruction Efficiency					
MCB, %	> 99.99972	> 99.99972 > 99.99972	> 99.99972	> 99.99972	%66.66
TCE, %	> 99.99995	> 99.99995	56666'66 <	> 99.99995	%66`66
Volatile Organics		es transm			
Semivolatile Organics	-	*****	-4-		See Section 6 for detailed test results
Chlorinated Dioxins and Furans ²	2.39E-13	9.63E-13	4.36E-11	1.49E-11	2.0E-7 g/sec
Nitrogen Oxides as NO2, lb/hr1	09'9	5.48	20.5	5.72	
Total Hydrocarbon as propane, lb/hr	660.0	0.099	660.0	0.099	uraves.

Carbon monoxide (CO), nitrogen oxides (NO_x) and total hydrocarbon (THC) individual test run averages from temporary CEM system used during the trial burn. CO results are corrected to 7% O₂.

Maximum TEQ emission rate.

Table 3-2

Summary of Process Operating Conditions During the Trial Burn - Mode A

Limit 464.81 NA NA 20 5.5 5.5 38.28 38.28 186,137 100 0.0 0.0 120 3 50 50	Parameter		Current		Mode A	le A		Proposed
nin 464.81 /min NA % 5.5 % 5.5 % 5.5 % 38.28 e, °F (max) 2,127 (proposed) 186,137 ppmv) 100 2O 0.0 1H ₂ O 3 ESP1 50 FSP7 50			Limit	Run 1	Run 2	Run 3	Average	Limit ²³
/min NA 20 20 % 5.5 e, °F (max) 2,127 (existing) 186,137 ppmv) 100 20 0.0 1H ₂ O 3 ESP1 50 ESP1 50	rdous Waste Feed Rate, Ibs/	/min	464.81	333.90	341.42	340,39	338.57	70 610
% 5.5 e, °F (max) 2,127 (existing) 186,137 ppmv) 100 20 0.0 1H ₂ O 0.0 1H ₂ O 3 ESP1 50	Spike Stream Feed Rate, 1b	s/min	NA	12.79	12.80	12.79	12.79	07.515
% 5.5 38.28 e, °F (max) 2,127 (existing) 186,137 (proposed) 186,137 ppmv) 100 20 0.0 140 1420 3 ESP1 50	et/Bag Feed Rate, per hour	(1	20	0	0	0	0	20
38.28 are, °F (max) 2,127 M (existing) 186,137 M (proposed) 186,137 (ppmv) 100 H ₂ O 0.0 in H ₂ O 3 ESP1 50 FSP2 50	mum Main Gas Blower SO2	% 5	5.5	8.36	8.26	9.03	8.55	5.5
emperature, °F (max) 2,127 y, ACFM (existing) 186,137 y, ACFM (proposed) 186,137 for CO. (ppmv) 100 essure, H ₂ O 0.0 °F 120 emister, in H ₂ O 3 ESP1 50 FSP2 50	nic Acid Production, ton/hr		38.28	28.52	27.93	28.00	28.15	38.28
y, ACFM (existing) 186,137 y, ACFM (proposed) 186,137 y, ACFM (proposed) 186,137 for CO. (ppmv) 100 essure, H ₂ O 0.0 P 120 emister, in H ₂ O 3 ESP1 50 FSD2 50	bustion Chamber Temperati	ıre, °F (max)	2,127	2150.94	2142.88	2146.29	2146.70	2146.70
y, ACFM (proposed) 186,137 for CO. (ppmv) 100 essure, H ₂ O 0.0 oF 120 emister, in H ₂ O 3 ESP1 50 FSP2 50	bustion Gas Velocity, ACFN	M (existing)	186,137	167,907	165,720	165,435	166,354	
for CO. (ppmv) 100 ressure, H ₂ O 0.0 PF 120 emister, in H ₂ O 3 ESP1 50 FSP2 50	bustion Gas Velocity, ACFI	M (proposed)	186,137	172,897	170,429	170,178	171,168	171,168
essure, H ₂ O 0.0 2p emister, in H ₂ O 3 ESP1 50 constant to the constant	ly Rolling Average for CO.	(hudd)	100	2.72	4.69	2.14	3.18	100
emister, in H ₂ O 3 ESP1 50	bustion Chamber Pressure, 1	H ₂ O	0.0	-1.41	-1.35	-1.37	-1.38	0.0
ESP1 50 50 FROD 50	Inlet Temperature, °F		120	60.06	86.07	89.33	88.50	120
ESP1 50 FSP2 50	ure Drop Across Demister,	in H ₂ O	. 3	16.47	16.19	16.31	16.32	3
ESP2 50	name to ECD VV	ESP1	50	67.77	67.85	68.22	67.95	50
27	rowel to ESF, N.v	ESP2	50	66.36	66.01	65.94	66.10	50

Buckets/Bags were not fed during the Trial Burn. £00

Proposed limits are based upon averages of Mode A and/or Mode B values where applicable. Proposed hazardous waste feed rate limit includes all waste feeds and spike streams.

Table 3-3

Summary of Process Operating Conditions during the Trial Burn - Mode B

Parameter		Current		Mode B	e B		Proposed
		Limit	Run 1	Run 2	Run 3	Average	Limit ^{2,3}
Hazardous Waste Feed Rate, Ibs/min	s/min	464.81	269.96	258.69	262.15	263.60	212.06
Total Spike Stream Feed Rate, I	lb/min	AN	11.55	11.55	11.55	11.55	07.516
Bucket/Bag Feed Rate, per hour	IL ₍₁₎	20	0	0	0	0	20
Minimum Main Gas Blower SO2, %	,2, %	5.5	8.41	9.56	8.42	8.80	5.5
Sulfuric Acid Production, ton/hr	ı	38.28	29.34	30.53	30.36	30.08	38.28
Combustion Chamber Temperature, °F (min)	ture, °F (min)	1884	68'6881	1869.13	1864,44	1874.32	1874.32
Combustion Gas Velocity, ACFM (existing)	M (existing)	186,137	144,309	135,561	133,579	137,816	
Combustion Gas Velocity, ACFM (proposed)	M (proposed)	186,137	148,269	139,241	137,446	141,652	171,168
Hourly Rolling Average for CO, (ppmv)	, (ppmv)	100	65.24	56.22	53.39	58.28	100
Combustion Chamber Pressure,	H ₂ O	0.0	62.1-	-1.14	-1.23	-1.39	0.0
ESP Inlet Temperature, °F		120	91.76	94.29	94.10	93.38	120
Pressure Drop Across Demister, in H2O	, in H ₂ O	3	14.98	14.82	14.88	14.89	3
Total Description	ESP1	50	69.14	89.69	69.55	69.46	50
I otal rower to ESF, N v	ESP2	. 05	70.82	73.10	71.70	71.87	50

Buckets/Bags were not fed during the Trial Burn.

Proposed limits are based upon averages of Mode A and/or Mode B values where applicable.

Proposed hazardous waste feed rate limit includes all waste feeds and spike streams.

01/19/2011

ECO SERVICES HOUSTON PLANT FLARE LOG SHEET

itation: 40 C	REGEN 2 SHU FR 264.1035(c)(FLARE START	8)		to the second se
	CFR 264.1035(c)(
		ME FAILURE?	T11.AT	DURATION
		NO _ NO	TIME TIME	DURATION DURATION
·	YES	NO _	TIME	DURATION
	YES	NO _	TIME	DURATION
tation: 40 C TIME A	FR 264.1035(c)(ABLE TO VEN FR 264.1035(c)(8	RATURE EVER 8) IT TO REGEN F		F(760 C)?YESNO
. DID FL itation: 40 C . TIME A	AME TEMPE FR 264.1035(c)(ABLE TO VEN	RATURE EVER 8) IT TO REGEN F	FURNACE: WAS FLARE IN S	

Revised 03/16/01 DBN

Attachment 2

Contained Vapor Combustor EPN 120 Log Sheet

Attachment 3

Contained Vapor Combustor EPN 170 Compliance Test Results

P. O. Box 890746 • Houston, Texas 77289-0746 • (281) 332-3118

SECTION 1.0 SUMMARY

Entech Engineering Inc. was retained by Rhodia Inc. to conduct an initial determination of compliance test of the Sulfuric Acid Regeneration Unit 2, Vapor Combustor 2 in Houston, Harris County, Texas. The primary objective of this program is to demonstrate performance of the Vapor Combustor 2 in controlling volatile organic compound (VOC) emissions according to the Texas Commission on Environmental Quality (TCEQ) Standard Permit No. 4802. The secondary objective of the program is to demonstrate initial compliance of the Vapor Combustor 2 in controlling Highly Reactive Volatile Organic Compound (HRVOC) emissions per the TCEQ Regulation V (30 TAC Chapter 115), Subchapter H. The emission performance test program was conducted on February 15 and 16, 2006 and coordinated by Mr. Floyd Dickerson and Mr. Craig Jongsma of Rhodia Inc. The TCEQ Region 12 (Houston) office was notified of the test program and Mr. Thomas Bill and Mr. Joseph Doby of that office were present during testing.

The Vapor Combustor 2 at the Rhodia Inc., Houston plant is an enclosed flare, manufactured by John Zink Company of Tulsa, Oklahoma, to control VOC's during normal tank breathing and railcar depressurizing. The vapor combustor 2 is designated in the permit as emission point number (EPN) 170. Pipeline-quality sweet natural gas is used as supplemental fuel. According to the permit, Vapor Combustor 2 has to conduct stack testing to demonstrate VOC emissions compliance (Special Condition 14). Testing occurred at the inlet of the vapor combustor to determine destruction and removal efficiency (DRE).

The compliance test was conducted during maximum production (loading) rates, which is identified as the combination of the maximum conditions as identified in the MAER as Vapor Combustor 2-Normal plus Vapor Combustor 2-Standby (maintenance). Those conditions included an outage on Regeneration Unit Number 2 Furnace (EPN 104), barge unloading of spent sulfuric acid into Tank 78 at a rate of 800 gallons per minute, working volume from spent sulfuric acid Tanks 48, 49 and 56, depressurization of six spent sulfuric acid railcars and depressurization of a spent sulfuric acid truck.

The vapor combustor 2 inlet offgas vent line is a 16-inch internal diameter vertical pipe. The upstream and downstream distances from the sampling plane meet the minimum unobstructed requirements of reference method 1. After destroying the emissions in the vapor combustor, the flue gas is vented to the atmosphere via 8-foot nominal internal diameter (ID) stack. The emission point for vapor combustor is located approximately 35 feet above grade.

According to the TCEQ Region 12 (Houston) office, the vapor combustor must demonstrate compliance with Standard Permit 4802 and the permit representation of 98 % destruction and removal efficiency (DRE). This program also determined compliance on highly-reactive volatile organic compound (HRVOC) emissions and determined HRVOC DRE based on ethylene mass emission rates. The initial compliance test comprised of inlet and outlet (stack) sampling for VOC

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and HRVOC testing by Reference Method (RM) 18. The vapor combustor inlet vent line velocity was measured by RM 2. Moisture at the inlet was determined based on the psychrometric condition of the gases.

Due to Rhodia Inc.'s safety guidelines preventing personnel from accessing the vapor combustor stack during operation, Entech personnel used the combined inlet flue gas composition (i.e., vent gas and natural gas) to determine the EPA stoichiometric Fd factor per the Reference Method (RM) 19 (40CFR60 Appendix A) and using the inlet (i.e., vent gas and natural gas) heat rates to determine the stack flue gas flow rates. The calculated stack flue gas flow rates were then used with measured stack concentrations to determine stack mass emission rates.

The mass emission rates at the inlet and stack were used to determine the DRE of the unit. The arithmetic average of the three runs was used to determine emission compliance test results at the maximum achievable operating condition. Process operational data were recorded by plant personnel to correlate the unit operating conditions to emission parameters.

A summary of the emission compliance test results in comparison to the regulatory requirements and permit specifications for the Vapor Combustor 2, EPN 170 are presented in Table 1. Test methods and equipment descriptions are presented in Section 2.0 and results and discussions are presented in Section 3.0.

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SECTION 3.0 RESULTS AND DISCUSSIONS

Entech Engineering conducted an initial determination of compliance test of the Sulfuric Acid Regeneration Unit 2, Vapor Combustor 2, EPN 170 at Rhodia Inc., Houston plant in Houston, Harris County, Texas. Sampling equipment was set up on February 14, 2006. Testing occurred on February 15 and 16, 2006.

For the emission tests, three 64-minute tests were conducted on one Vapor Combustor 2 to measure inlet vent line, natural gas fuel line and stack VOC and HRVOC emissions while operating at the maximum achievable rate. Inlet pollutant and miscellaneous gas compositions along with flow rates were used to calculate by RM 19 the stack flow rate. The inlet and stack mass emission rates were used to calculate destruction and removal efficiency of the unit. Summaries of the emission compliance test results are presented in Tables 2 through 6. At the stack no VOC concentrations were measured by the gas chromatograph except propane in Test ID 1, therefore the method detection limit for all other VOC and HRVOC species analyzed for is used to represent the stack emission concentration and mass emission.

Other pertinent data of the test program is contained in the appendices. The field raw data is contained in Appendix A. Laboratory data and data calculations are presented in Appendices B and C. Instrument specifications, equipment calibrations, process data, resumes, chain of custody, and personnel information are presented in Appendices D through J.

P. O. Box 890746 . Housian, Texas 77289-0746 . (281)332-3118

Table 2.

Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 170) VOC Emission Performance Test Summary Houston, Harris County, Texas

	T	I 		
TestID		2	3	Аусгадс
Date	02/15/06	02/16/06	02/16/06	
Time .	11:15 - 12:19	10:05 - 11:10	14:40 - 15:41	<u> </u>

VOC Emission Data

_					
Inict Waste Gas Line	(ib/hr)	12,891	5.714	4.401	7.669
Fuel Gas Line	(lb/hr)	2.518	12.611	11.909	12.346
Combined Inlet Waste Gas and Fuel Gas Line VOC's	(lb/hr)	25,409	18.325	16.310	20.014
Stack VOC	(lb/hr)	0.067	0.063	0.066	0.065
VOC DRE	(%)	99.74%	99.66%	99.59%	99.7%

HRVOC Emission Data *

Inlet Waste Gas Line Ethylene	(lb/lu)	0.0724	0.2495	0.2124	0.1781
Stack Ethylene	(lb/lu)	0.0024	0.0023	0.0024	0,0024
HRVOC DRE	(%)	96,64%	99.07%	98.85%	98.2%

Stack HRVOC**	(1b/hr)	0.0253	0.0241	0.0255	0.0249

^{*}Note: Ethylene was the only HRVOC measured by the Gas Chromatograph at the infel. The gas chromatograph analysis at the stack resulted in no measurable HRVOC, therefore the method detection limit (MDL) for ethylene is used at the stack. The HRVOC DRE (destruction and removal efficiency) was determined based on ethylene mass emission rates.

^{**}Note: The gas chromatograph analysis at the stack resulted in no measurable HRVOC, therefore the toethod detection limit (MDL) for all HRVOC's is used at the stack.

P. O. Box 890746. Houston, Texas 77289-0746. (281)332-3118

Table 3.

Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 170)

VOC Emission Performance Test Summary Houston, Harris County, Texas

Test ID	1	2	3
Date	02/15/06	02/16/06	02/16/06
Time	11:15 - 12:19	10:05 - 11:10	14:40 - 15:41

Inlet Waste Vent Line Parameters

Inlet Waste Gas Flow Rate	(SCFM)	871.99	1241.67	853.25
	(SCM/hr)	1481.69	2109.85	1449.84
Inlet Waste Gas HHV	(Btu/SCF)	5.37	1.68	1.93
	(Btu/lb)	73.00	23.00	26.00

Combined Inlet Waste Vent Gas Line and Fuel Gas Line Parameters

Fd Factor	(DSCF/MMBtu)	12273.14	13497.09	12434.92	
Total Heat Input	(MMBtu/hr)	12.20	12.67	11.67	
Inlet Waste Gas Heat Input	(MMBtu/br)	0.28	0.13	0.10	
Fuel Gas Heat Input	(MMBtu/hr)	11.92	12,55	11.57	
	(Btw/lb)	21919	21902	21902	
Fuel Gas HHV	(Btu/SCF)	997.81	995.51	995.51	
Fuel Gas Flow Rate	(SCFM)	199.15	210.07	193.73	

Stack Parameters

Flue Gas Flow Rate*	(DSCFM)	6248.26	5952.04	6287.47
Oxygen Content	(%)	12.55	10.89	12.86
Carbon Dioxide Content	(%)	3.50	4.25	3,51

^{*} The flue gas flow rates were determined by Reference Method 19 Fd factor.

Table 4,

Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 178) Stack Performance Test Summary Houston, Harris County, Texas

Tost 1D	1	2	3
Date	02/15/06	02/16/08	02/16/06
Time	13:15 - [2:19	10:05 - 11:10	14:40 - 15:41

Stack Gas Parameters

Stack Flow Rate	(SCFM)	6248.26	5952.04	6287.47
	(SCM/hr)	30617,04	10113.70	10683,66
Suct Mol. We	lb/mote	25.870	26,260	25,760
Oxygen Content	(%)	12.55	10.09	12.86
Carbon Dioxide Content	(%)	3.50	4.25	3,51
Nitrogen Content	(%)	82.21	83,92	83.88
Moisture Content	(%)	1.74	1.74	1.74

Stack Volatile Organic Compounds (VOC) Gas Data

Component Name	Molecular Formula	Concentration**	Mass Rato Ib/Iv	Concentration**	Mass Rate Ib/hr	Concentration**	Mass Rate Ib/hr
Methane	16 04	0.2770	0.0043	0.0000	0.0000	0.0000	0.0000
Ethane	30,07	0.1300	0.0038	0.1710	0.0048	0.4200	0.0124
Ethylene	28.05	0.0890	0.0024	0.0890	0,0923	0.0890	0.0024
Propane	44.09	01110	0.0048	0.0880	0.0036	0.0380	0.0038
Propylene	42.08	0,0870	0.0036	0,0870	0.0034	0.0870	0,0036
Isobutane	58.12	0.0710	0.0040	0.0710	0.0038	0.0710	0.0040
n-Bulenc	58.12	0.880.0	0.0050	0.0860,0	0.0047	0.0880	0.0050
cis 2-Butene	56,10	0,0500	0.0027	0.0500	0.0026	0.0500	0.0027
1-Butens	56,10	0,0900	0.0049	0.0900	0.0047	0,0900	0.0049
Isobutylene	56,10	0 0910	0.0050	0.0910	0,0047	0.0910	0.0050
trans 2-Butene	56.10	0.0320	0.0017	0.0320	0.0017	0.0320	0.0018
n-Pentané	72.15	0.0940	0.0056	0.0940	0.0063	0.0940	0.0066
1,3-Butadiene	54.09	0.0940	0.0049	0.0940	0.0047	0.0940	0,0050
I-Pentene	70.13	0.0860	0.0059	0,0860	0.0056	0.0860	0.0059
n-Hexane	86.17	0.1000	0.0084	0.1000	0.0080	0.000	0,0084
1-liexent	\$4.15	0.0830	0.0058	0.0830	0.0065	0.0830	0,0068
Total VOC Stack ***		1.1660	0.0667	1.1430	0.0626	1.1430	0.0661
Total HRVOC Stack		0.5330	0.0253	0.5330	0.0241	0.5330	0.0255

^{*} Mote: Total HRVOC includes ethylene, propylene, cis 2-butene, trans 2-butene, 1-butene, isobutylene, and 1,3-butadiene.

^{**} Note: No VOC concentrations were measured by the gas chromatograph except propane in Test ID 1, therefore all other VOC concentrations are presented as Method Detection Limit (MDL) in parts-per-million by volume.

^{***} Note: Total VOC does not include Methane and Ethant.

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Table 5.

Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 170) Inlet Waste Vent Line Performance Test Summary Houston, Harris County, Texas

Test ID	I	2	3	
Date	02/15/06	02/16/06	02/16/06	
Time	11:15 - 12:19	10:05 - 11:10	14:40 - 15:41	

Inlet Waste Vent Line Parameters

			1	I
Duct Temperature	(deg. F)	84.06	\$2.56	B7.06
Vent Gas Velocity	(ft/sec)	10.60	15,05	10,47
Vent Gas Flow Rote	(SCFM)	871.99	1241.67	853.25
	(SCM/hr)	1481.69	2109.85	1449.84
Vent Mol. Wt.	lb/mole	27.847	27.803	27,710
Oxygen Content	(% dry)	3.10	3.54	2,65
Carbon Dioxide Content	(% dry)	0,30	0.00	0.00
Nitrogen Content	Nitrogen Content (% dry) 92.5		92.69	92.98
Moisture Content	(%)	3.90	3.71	4.30

Inlet Waste Vent Line Volatile Organic Compounds (VOC) Gas Data

-	Traste Tent L	1	7.541114 60	in pounts (100,000		r
Component Name	Molecular Formula	Volume % wet	Mass Rate	Volume % wet	Mass Rate	Volume % wet	Mass Raie
Methane	16.04	0,0003%	0.007	0.0003%	0.009	0.0003%	0.006
Ethane	30.07	0.0001%	0.004	0.0001%	0.006	0.0001%	0.004
Ethylene	28.05	0.0019%	0.072	0.0046%	0.249	0.0057%	0.212
Propane	44.09	0.0518%	3.101	0.0222%	1.893	0.0217%	1.271
Propylene	42.08	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Isobutane	58.12	0.0267%	2.107	0.0038%	0,427	0.0047%	0.363
n-Butane	58.12	0.0921%	7.268	0.0271%	3.045	0.0320%	2,471
cis 2-Butena	\$6.10	0,0000%	0.000	0,0000%	0.000	0.0000%	0.000
1-Butenc	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Isobutylene	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
trans 2-Butene	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
n-Pentane	72.15	0.0002%	0.020	0.0001%	0,014	0.0001%	0.010
1,3-Butadiene	54.09	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
)-Pentene	70.13	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
n-Hexane	86.17	0.0001%	0.012	0.0000%	0.000	0.0000%	0.000
1-Hexene	84.16	0.0001%	0.011	0.0000%	0.000	0.0000%	0.000
Unknown as C3**	44.09	0.0050%	0,299	0.0010%	0.085	0.0012%	0.070
Total VOC Vent Gas *	lb/hr	<u> </u>	12.891		5.714		4.401

^{*} Note: Total VOC does not include methane and ethane.
** Note: All unspeciated VOC's are presented as propane equivalent.

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Table 6.

Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 170) Fuel Gas Line Performance Test Summary Houston, Harris County, Texas

			1
Test ID	<u> </u>	2	3
Date	02/15/06	02/16/06	02/16/06
Time	11:15 - 12:19	10:05 - 11:10	14:40 - 15:41

Fuel Gas Line Parameters

Vent Gas Flow Rate	(\$CFM)	199.15	210.07	193.73
	(SCMAr)	338.40	356.95	329.19
Vent Mol. Wt.	lb/mole	17.220	17.200	17,200
Oxygen Content	(% dry)	0.14	0,21	0.1 B
Carbon Dioxide Content	(% dry)	1.65	1.51	1.58
Nitrogen Content	(% dry)	0.89	1.06	0.97
Moisture Content	(%)	1.74	1.74	1.74

Fuel Gas Line Volatile Organic Compounds (VOC) Gas Data

Component Name	Molecular Formula	Volume % wet	Mass Rate lb/hr	Volume % wet	Mass Rate	Volume % wet	Mass Rate
Methane	16.04	92.8420%	461.85]	92.8260%	487.084	92.8340%	449.250
Ethanc	30.07	1.9800%	18.462	1.9200%	18.883	1.9500%	17,687
Ethylene	28.05	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Propane	44.09	0.4480%	6.126	0.4200%	6.058	0,4340%	5.773
Propylene	42.08	0.0000%	0,000	0.0000%	0.000	0.0000%	0.000
Isobutane	58.12	0.1060%	1.910	0.1030%	1.958	0.1050%	1.841
n-Butane	58.12	0.0980%	1.766	0.0950%	1.806	0.0970%	1.701
cis 2-Butene	56.10	0.0000%	0,000	0.0000%	0.000	0.0000%	0.000
1-Butene	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Isobutylene	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0,000
trans 2-Butene	56.10	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
n-Pentane	72.15	0.0270%	0.604	0.0270%	0.637	0.0270%	0.588
1,3-Butadiene	54.09	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Isopeniane	72.15	0.0490%	1.096	0.0470%	1.109	0.0480%	1.045
n-Hexane	86.17	0.0170%	0.454	0.0160%	0.451	0.0160%	0.416
1-Hexene	84.16	0.0000%	0.000	0.0000%	0.000	0.0000%	0.000
Unknown as C6	86.17	0.0210%	0,561	0.0210%	0.592	0.0210%	0.546
					<u> </u>	<u> </u>	
	lb/hr	}	12.518	1	12.611	<u> </u>	11.909

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Table 7. Rhodia Inc., Houston, Texas Vapor Combustor 2 (EPN 170) Process Data Summary Houston, Harrls County, Texas

Test ID '	1	2	3
Date	02/15/06	02/16/06	02/16/06
Time	11:15 - 12:19	10:05 - 11:10	14:40 - 15:41
Stack Temperature (F)	1522.0	1528.8	1536,2
Natural Gas Flow (SCFM)	199.2	210.1	193.7
Venturi Scrubber (pH)	13,1	12.4	12.4
Packet Column Scrubber (pH)	10.6	10.7	12.1
Pressure Scrubber Venturi Injet (in WC)	-0.6	-0.4	-0.4
Pressure Scrubber Column Inlet (in WC)	4.3	5.1	5.1
Pressure Vapor Combustor inlet (in WC)	1.5	2.0	1.5

Note: Data presented is average of at least four 15 minute readings. Raw process data is presented in Appendix E.

Attachment 4

Baton Rouge Contained Vapor Combustor Compliance Test Results

VOLATILE ORGANIC CARBON DESTRUCTION REMOVAL EFFICIENCY OF COMBUSTOR

CONDUCTED ON SEPTEMBER 23, 2003

Prepared for

RHODIA, INC.

1275 Airline Highway Baton Rouge, Louisiana 70807

Prepared by

SETCO

SCHWARTZ ENVIRONMENTAL TESTING COMPANY, INC. P.O. Box 1667 Prairieville, Louisiana 70769

Project Number 03-107

1.0 INTRODUCTION

Setco conducted emission testing of the acid plant vapor combustor (combustor) for Rhodia, Inc. (Rhodia) at the manufacturing facility located in Baton Rouge, East Baton Rouge Parish, Louisiana. The test was conducted to determine Volatile Organic Carbon (VOC) destruction removal efficiency between the inlet and exhaust stack. Additionally, the exhaust gas stream was measured for sulfur dioxide (SO₂), oxides of nitrogen (NO₄), and carbon monoxide (CO) emission rates. The combustor is identified as Point I.D. Number 27 in the Authorization to Construct issued October 28, 2002.

The test series of the combustor unit was conducted by Setco in accordance with EPA Reference Methods outlined in New Source Performance Standards (NSPS) of Appendix A, Title 40, Part 60 of the <u>Code of Federal Regulations</u> (40 CFR 60). VOC emission samples from the inlet vent and exhaust stack were collected by EPA Reference Method 18. Measurement and determination of flow rate parameters from the inlet vent and exhaust stack were conducted by EPA Reference Methods 1-4. O₂/CO₂₁ SO₂₂, NO₂₃, and CO were measured with continuous emission monitors by EPA Reference Methods 3A/3A, 6C, 7E, and 10, respectively.

The emission testing series was conducted by Scott Neumann, Anthony Glass, Keith Delk, and Miles Holley of Setco. Test coordination was managed by John Richardson of Rhodia. The test series was not witnessed by a LDEQ representative, although LDEQ was informed of the test date.

2.0 SUMMARY OF TEST RESULTS

The combustor (EIQ Number 27) is permitted for maximum/average SO₂, NO_x, and CO emission rates of 0.045/0.001, 4.67/0.373, and 6.22/0.497 lb/hr, respectively. Continuous emission monitoring of O₂/CO₂, SO₃, NO₄, and CO consisted of 3, 1-hour runs conducted during 750-850, 925-1025, and 1055-1155, respectively. Collection of VOC samples consisted of 3, 30-minute test runs conducted during 800-830, 930-1000, and 1100-1130, respectively.

Table 2-1 presents a summary of SO₂, NO₃, and CO emission test results. The average SO₂, NO₃, and CO mass emission rate were determined to be 0.03, 0.96, and 3.09 lb/hr, respectively. Table 2-2 presents the VOC emission test results. The average VOC mass emission rates entering and exhausting from the combustor were measured to be 38.51 and 0.76 lb/hr, respectively. Based on these values, the average VOC removal efficiency was calculated to be 98.0%. The average VOC mass emission rate of 0.76 lb/hr is below the maximum permit limitation of 31.583 lb/hr.

In Conclusion: The average SO₂, NO₃, CO, and VOC mass emission rate in units of lb/hr are below the maximum allowable limitation of the Authorization to Construct. VOC destruction removal efficiency was determined to be greater than the 95% minimum limitation required by NSPS Subpart Kb.

SO₂, NO₃, CO, and VOC mass emission rates were determined by Setco from "dry" basis concentration units of ppm, as measured with Reference Method CEMS; and, stack gas volumetric flow rate, corrected to dry standard conditions (29.92 in. Hg, 528°R). A summary of the stack gas flow rate parameters measured during the test series is presented in Table 2-3.

VOC emission concentration was determined from the laboratory analysis of the collected integrated samples by gas chromatography. The gaseous integrated samples were analyzed for $C_1 - C_7$, aliphatic hydrocarbons constituents against known calibration standards. All raw test data and calculations are contained in the Appendix.

TABLE 2-1

EMISSION TEST RESULTS, COMBUSTOR EXHAUST, RHODIA, INC., BATON ROUGE, LOUISIANA, SEPTEMBER 23, 2003

PARAMETER	RUN I	RUN 2	RUN 3	average		
Test Date	9/23/2003	9/23/2003	9/23/2003	N/A		
Test Time	750-850	925-1025	1055-1155	N/A		
	Emission Test R	lesults				
SO, Concentration, ppm	0.79	0.88	0.84	0.84		
SO, Mass Emission Rate, lh/hr	0.03	0.04	0.03	0.03		
SO ₁ Maximum Permitted Mass Emission Rate, lb/hr						
NO, Concentration, ppm	31.08	30.70	37.45	33.08		
NO, Mass Emission Rate, ib/hr	0.90	0.89	1.10	0.96		
NO, Maximum Permitted Mass Emission Rate, lb/hr						
CO Concentration, ppm	222.15	207.81	95.46	175.14		
CO Mass Emission Rate, lb/hr	3.90	3,67	1.71	3.09		
CO Maximum Permitted Mass Endssi	6,22					
O, Concentration, %	13.46	14.45	13.07	13,66		

Source: Selco, 2003

TABLE 2-2

EMISSION TEST RESULTS, COMBUSTOR, RHODIA, INC., BATON ROUGE, LOUISIANA, SEPTEMBER 23, 2003

PARAMETER	RUN 1	RUN 2	RUN 3	AVERAGE	
Test Date	9/23/2003	9/23/2003	9/23/2003	N/A	
Test Time	800-830	930-1000	1100-1130	N/A	
VO	C Emission Tes	l Results			
Inict VOC ¹ Mass Flow Rate, lb/hr	35,53	33.52 -	46,49	38.51	
Exhaust VOC1 Mass Flow Rate, lb/hr	0.91	0.67	0.70	0,76	
EIQ Maximum Permitted VOC ¹ Mass Flow Rate, lb/hr					
VOC1 Destruction Removal Efficiency2, %	97.4	98.0	98.5	98.0	
NSPS Subpart Kb Minimum VOC¹ Destruction Removal Efficiency, %					
Operational Data					
Firebox Temperature, °F	1549	1561	1561	1557	

Source: Selco, 2003

Notes:

VOC - Volatile Organic Carbon compounds (total hydrocarbons excluding methane and ethane) Destruction Removal Efficiency = (Inlet - Outlet)/(Inlet) × 100%.

TABLE 2-3

FLOW RATE PARAMETERS, COMBUSTOR, RHODIA, INC., BATON ROUGE, LOUISIANA, SEPTEMBER 23, 2003

PARAMETER	RUN 1	RUN 2	RUN 3	AVERAGE		
Inlet Gas Stream						
Stack Gas Temperature, °F	. 80,2	87.4	99.3	89.0		
Stack Gas Temperature, °R	540.2	547.4	559.3	549,0		
Moisture Content ¹ , % volume	4.10	3.74	6.83	4.89		
Velocity, fVs	7.67	7.94	8.28	7.96		
Volumetric Flow Rate, 113/min	253.1	262.0	273.2	262.8		
Volumetric Flow Rate ¹ , ft³/min	237.3	243.3	240.7	240.4		
Outlet Gas Stream						
Stack Gas Temperature, °F	1385,1	1322,2	1335,7	1347.7		
Stack Gas Temperature, "R	1845.1	1782.2	1795.7	1807.7		
Moisture Content, % volume	8.23	8.67	9.44	8.78		
Velocity, fVs	16.40	15.98	16.44	16,27		
Volumetric Flow Rate, 13 ¹ /min	15360,2	14966,9	15397.7	15241.6		
Volumetric Flow Rate ¹ , ft ³ /min	4031.0	4046.8	4102.7	4060.2		

Source; Setco, 2003

Notes: .

Corrected to dry standard conditious.

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COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

CERTIFIED MAIL: Return Receipt Requested (7015 3010 0000 3182 3806)

July 20, 2016

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

April 1, 2016 to June 30, 2016 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations Corp. in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact David Laurie at (713) 924-1484 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Eco Services Operations Corp. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: <u>June 30, 2016</u>

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)	1	
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial	•	,
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2	•	
industrial furnace, including the vapor	. ,	
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)	*	
Daily inspections of control device continuous	x Yes	7.67
monitoring data (temperature of TS vapor	Except as Noted	Į
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

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Eco Services Operations Corp. Houston Plant

USEPAZOALLAS, TX ASSOCIATE DIRECTOR

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COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

Certified Mail; Return Receipt Requested (7015 3010 0000 3182 3813)

July 20, 2016

Mr. Jeff Robinson Chief, Air Permits Section 6PD-R U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

HON Semiannual Report per 40 CFR 63.152(c)

TCEQ Identification Nos.: RN100220581/CN604683482

Dear Mr. Robinson:

Eco Services Operations Corp. (Eco Services), formally Eco Services Operations LLC, is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Eco Services submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Eco Services submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of January 1 to June 30, 2016.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

HON Group 1 wastewater and residuals may be stored in one or more of the six (6) Treatment Services (TS) tanks. There were no control equipment failures identified for these tanks in the reporting period.

63.146(d) - Treatment process monitoring data.

The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

Eco Services Operations Corp. Houston Plant 8615 Manchester Street Houston, TX 77012 63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

The TS tanks normally vent to the Regeneration Unit No. 2 (Regen 2) sulfuric acid furnace which is exempt from monitoring per 63.139(d)(4)(iv). In the event that Regen 2 is unavailable, tank vapors are routed to the Treatment Services Vapor Combustor (TSVC). The TSVC minimum combustion temperature is 1,500°F per operating permit. During this reporting period, there were no instances of the daily average TSVC combustion temperature being under 1,500°F while TS tanks were venting to it. There were no days when insufficient monitoring data were collected during this reporting period.

63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) - For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

63.146(e)(1), Table 20 item (8)(ii) and 63.148(j)(3) - Report the times and durations of any periods when the bypass valves are moved to the diverting position, the seal has been changed, the seal mechanism is broken, or the key to unlock the bypass line valve was checked out.

There were no bypass valve or car-seal abnormalities this reporting period. We do not use lockand-key mechanisms for this purpose.

Please contact David Laurie at 713-924-1484 if you have any comments or require any additional information on this matter.

Sincerely,

In elling in Growill William McConnell

Plant Manager

Air Section Manager, TCEQ, Region 12 cc:

Director, Health and Human Services Department, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

153215 28 11 0000460901

US EPA, DALLAS, TX ECOSERVICES ASSOCIATE DIRECTOR

16 AUG -5 AM 9: 07

Eco Services LLC Houston Plant

COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

July 31, 2016

Al An Ico

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Subject:

Eco Services LLC

Houston, Texas Plant

NSPS Kb Semiannual Report - 1st Half 2016

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS)	liquids (VOL)	Vapor Combustor (TSVC) as backup
	Tanks		
Tk 48	Spent Acid	Spent sulfuric	Regeneration Unit No.2 Furnace with Spent
Tk 49	(SA)Tanks	acid with	Acid Vapor Combustor as backup.
Tk 53		potential for	
Tk 56*		containing	
Tk 78*		volatile organic	
		liquids	

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.

Eco Services LLC Houston Plant 8615 Manchester Street Houston, TX 77012



Spent Acid Tanks Summary Report

Pollutant	VOC
Reporting period dates:	1/1/2016 to 6/30/2016
Company:	Eco Services LLC Houston site
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Spent Acid Tank Farm
Total source operating time in reporting period:	4,344 hours
Duration of excess emissions in reporting period due to:	
a. Startup/shutdown	0.0 hours
b. Control equipment problems	0.00 hours
c. Process problems	516.23 hours
d. Other known causes	0.0 hours
e. Unknown causes	0.0 hours
Total duration of excess emission	516.23 hours
Total duration of excess emissions	11.8 %



Eco Services LLC Houston Plant

TS Tanks Summary Report

Pollutant	VOC					
Reporting period dates:	1/1/2016 to 6/30/2016					
Company:	Eco Services LLC Houston site					
Emission Limitation:	22.22 lbs/hr when venting to TSVC					
Address:	8615 Manchester Houston, TX 77012					
Monitor Manufacturer and Model No:	Not Applicable					
Date of Latest CMS Certification or Audit:	Not Applicable					
Process Unit Description:	Treatment Services Tank Farm					
Total source operating time in reporting period:	4,344 hours					
Duration of excess emissions in reporting period due to:						
f. Startup/shutdown	0 hours					
g. Control equipment problems	0 hours					
h. Process problems	0.0 hours					
i. Other known causes	0.0 hours					
j. Unknown causes	0 hours					
Total duration of excess emission	0.0 hours					
Total duration of excess emissions	0.0 %					

If you have any questions concerning this matter, please call David Laurie at (713) 924-1484.

Sincerely,

William McConnell

Plant Manager Solvay USA Inc.

Attachment

Eco Services LLC Houston Plant 8615 Manchester Street Houston, TX 77012



Eco Services LLC Houston Plant

Cc:

Executive Director, MC-109
Texas Commission on Environmental Quality
P.O. Box 13087
Austin, TX 78711-3087

Air Section Manager Texas Commission on Environmental Quality 5425 Polk Avenue, Suite H Houston, TX 77023-1486

Bureau Chief Bureau of Air Quality Control City of Houston 7411 Park Place Blvd. Houston, TX 77087-4441

Director
Harris County Public Health and Environmental Services
Environmental Public Health Division
101 S. Richey Suite G
Pasadena, TX 77506

Eco Services LLC Houston Plant 8615 Manchester Street Houston, TX 77012 53215 V&

0000460901



Houston Plant

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 1699)

May 3, 2016

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

January 1, 2016 to March 31, 2016 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations LLC in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank vent stream is introduced into the primary control device flame zone, the SARU industrial furnace.

• Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the average temperature of the gas stream in the combustion zone for the TS Vapor Combustor was <50°F below design temperature when being used as the control device for the TS storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell
Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

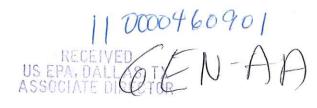
Eco Services Operations LLC Houston, Texas Benzene Waste NESHAP Inspection Requirements For Overtally Paried Frediens Manual, 21, 2016

For Quarterly	Period	Ending:	March	31, 2016

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	-
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	***************************************
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	-
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

45-3215 V8





Eco Services Operations Corp. Houston Plant 16 NOV -4 PM 2: 47

COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

CERTIFIED MAIL: Return Receipt Requested (7015 3010 0000 3182 3868)

October 27, 2016

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733 AI/AI/CO

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

July 1, 2016 to September 30, 2016 EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations Corp. in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations LLC hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

Eco Services Operations Corp. Houston Plant 8615 Manchester Street Houston, TX 77012 Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact me at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Eco Services Operations Corp. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: September 30, 2016

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		***************************************
Regeneration Unit No. 2 industrial furnace) per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

45 3215 VF



Eco Services Operations Corp. Houston Plant RECEIVED US EPA, DALLAS, TX ASSOCIATE DIRECTOR

17 APR - 3 PM 4: 15

COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

CERTIFIED MAIL: Return Receipt Requested (7015 1520 0003 4945 8385)

April 27, 2017

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733 AI/AI/CO

FRS 116000460901

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report January 1, 2017 to March 31, 2017

EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations Corp. in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations Corp. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

• Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the average temperature of the gas stream in the combustion zone for the TS Vapor Combustor was <50°F below design temperature when being used as the control device for the TS storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

William McConnell Plant Manager

cc:

Air Section Manager, TCEQ, Region 12

Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Eco Services Operations Corp. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: March 31, 2017

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)		
Initial and quarterly visual inspections of	Except as Noted x Yes	
containers per 61.345(b)		
Annual Method 21 inspections of treatment	Except as Noted	
system openings (Regeneration Unit No. 2)	x Yes	
per 61.348(e)(3)(ii)	Except as Noted	
Annual Method 21 inspections of closed vent		
systems (from tanks to TS vapor combustor	x Yes	
and Regeneration Unit No. 2 industrial	Except as Noted	ļ
furnace) per 61.349(a)(1)(i)]	
Quarterly visual inspections of closed vent		
systems and control devices (C	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
ndustrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
ndustrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
nonitoring data (temperature of TS vapor	Except as Noted	
ombustor and "selected parameter" on	•	
Regeneration Unit No. 2 industrial furnace)		
er 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.





17 JAN 11 AM 11: 04

Certified Mail: Return Receipt Requested (7011 2000 0001 4575 4898) & ENFORCEMENT DIV.

January 5, 2017

Compliance Assurance and Enforcement Division (6EN) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

AI/AI/CO

Subject:

Eco Services Operations Corp.

Houston, Texas Plant

NSPS Kb Semiannual Report - 2nd Half 2016

Permit No. O-03049

To Whom It May Concern:

Per the 40 CFR Part 60 (NSPS) Subpart Kb operating plan for the Houston plant, the following tanks are subject to vapor control, or assumed to be subject to vapor control, per NSPS Subpart Kb.

Tank No.	Description	Contents	Control Device
B-1	Treatment	Volatile organic	Regeneration Unit No.2 Furnace with TS
B-2	Services (TS) Tanks	liquids (VOL)	Vapor Combustor (TSVC) as backup
Tk 48 Tk 49 Tk 53 Tk 56* Tk 78*	Spent Acid (SA)Tanks	Spent sulfuric acid with potential for containing volatile organic liquids	Regeneration Unit No.2 Furnace with Spent Acid Vapor Combustor as backup.

^{*}Available information indicates that tanks 56 and 78 haves not been reconstructed or modified since 1984, but are listed for completeness.

40 CFR 60.7 requires a semiannual report for these tanks.

Spent Acid Tanks Summary Report

Pollutant	VOC
Reporting period dates:	8/1/2016 to 12/31/2016
Company:	Eco Services Houston site
Emission Limitation:	25.93 lbs/hr when venting to secondary APVC (vapor combustor)
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CEMS Certification or Audit:	Not Applicable
Process Unit Description:	Spent Acid Tank Farm
Total source operating time in reporting period:	4,416 hours
Duration of excess emissions in reporting period due to:	
a. Startup/shutdown	0.0 hours
b. Control equipment problems	0.00 hours
c. Process problems	120.83 hours
d. Other known causes	0.0 hours
e. Unknown causes	0.0 hours
Total duration of excess emission	120.83 hours
Total duration of excess emissions	2.74 %

TS Tanks Summary Report

Pollutant	VOC
Reporting period dates:	8/1/2016 to 12/31/2016
Company:	Eco Services Houston site
Emission Limitation:	22.22 lbs/hr when venting to TSVC
Address:	8615 Manchester Houston, TX 77012
Monitor Manufacturer and Model No:	Not Applicable
Date of Latest CMS Certification or Audit:	Not Applicable
Process Unit Description:	Treatment Services Tank Farm
Total source operating time in reporting period:	4,416 hours
Duration of excess emissions in reporting period due to:	
f. Startup/shutdown	0 hours
g. Control equipment problems	0 hours
h. Process problems	80.0 hours
i. Other known causes	0.0 hours
j. Unknown causes	0 hours
Total duration of excess emission	80.0 hours
Total duration of excess emissions	1.81 %

If you have any questions concerning this matter, please call Floyd Dickerson at (713) 924-1408.

pullen Jin-correll

Sincerely,

William McConnell Plant Manager

> Eco Services Corp. Houston Plant 8615 Manchester Street Houston, TX 77012

Cc:

Executive Director, MC-109 Texas Commission on Environmental Quality P.O. Box 13087 Austin, TX 78711-3087

Air Section Manager Texas Commission on Environmental Quality 5425 Polk Avenue, Suite H Houston, TX 77023-1486

Bureau Chief Bureau of Air Quality Control City of Houston 7411 Park Place Blvd. Houston, TX 77087-4441

Director
Harris County Public Health and Environmental Services
Environmental Public Health Division
101 S. Richey Suite G
Pasadena, TX 77506

Eco Services Corp. Houston Plant 8615 Manchester Street Houston, TX 77012 45 3215 V8 116000460901



Eco Services Operations Corp. Houston Plant

RECEIVED US EPA, DALLAS, TX ASSOCIATE DIRECTOR

17 JAN 27 PM 2: 59

COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

Certified Mail; Return Receipt Requested (7011 2000 0001 4575 4935)

January 24, 2017

Mr. Jeff Robinson Chief, Air Permits Section 6PD-R U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

HON Semiannual Report per 40 CFR 63.152(c)

TCEQ Identification Nos.: RN100220581/CN604683482

Dear Mr. Robinson:

Eco Services Operations Corp. (Eco Services), formally Eco Services Operations LLC, is an offsite treatment facility for 40 CFR Part 63 Subpart G (HON) Group 1 wastewater streams and residuals. Eco Services submitted a letter on August 6, 1998 certifying that it will manage and treat any HON-regulated Group 1 wastewater stream or residual removed from a Group 1 wastewater stream in accordance with the applicable requirements in 40 CFR 63.133 through 63.147. On November 8, 2006, Eco Services submitted the Notification of Compliance Status (NCS) Report per 40 CFR 63.152(b). Per 40 CFR 63.146(c) and 63.152(c), semiannual reports are also required. This submittal includes the semiannual report for the period of July 1 to December 31, 2016.

Specific elements of the semi-annual report are listed below:

63.146(c) - For each tank storing HON Group 1 wastewater or residuals, the results of each inspection in which a control equipment failure (a gasket, joint, lid, cover, or door has a crack or gap, or is broken) was identified. Include the date of the inspection, identification of each waste management unit (tank) in which a control equipment failure was detected, description of the failure, and description of the nature of and date the repair was made.

HON Group 1 wastewater and residuals may be stored in one or more of the six (6) Treatment Services (TS) tanks. There were no control equipment failures identified for these tanks in the reporting period.

63.146(d) - Treatment process monitoring data.

The treatment process is a RCRA unit and is exempt from monitoring per 63.138(h).

Eco Services Operations Corp. Houston Plant 8615 Manchester Street Houston, TX 77012 63.146(e)(1), Table 20 item (1) - For each tank storing HON Group 1 wastewater or residuals that vents to a thermal incinerator for vapor control, report all daily average temperatures that are outside the range established in the NCS or operating permit and all operating days when insufficient monitoring data are collected.

The TS tanks normally vent to the Regeneration Unit No. 2 (Regen 2) sulfuric acid furnace which is exempt from monitoring per 63.139(d)(4)(iv). In the event that Regen 2 is unavailable, tank vapors are routed to the Treatment Services Vapor Combustor (TSVC). The TSVC minimum combustion temperature is 1,500°F per operating permit. During this reporting period, there were no instances of the daily average TSVC combustion temperature being under 1,500°F while TS tanks were venting to it. There were no days when insufficient monitoring data were collected during this reporting period.

63.146(e)(1), Table 20 item (8)(i) and 63.148(j)(2) — For closed vent systems used to convey HON wastewater tank vapors to a control device, any bypass valves and lines must be equipped with a flow indicator or car-seal. Report the times and durations of all periods when the vent stream is diverted through a bypass line or the monitor (flow indicator) is not operating.

The vent stream was not diverted to the atmosphere this reporting period. We do not use flow indicators for this purpose.

63.146(e)(1), Table 20 item (8)(ii) and 63.148(j)(3) - Report the times and durations of any periods when the bypass valves are moved to the diverting position, the seal has been changed, the seal mechanism is broken, or the key to unlock the bypass line valve was checked out.

There were no bypass valve or car-seal abnormalities this reporting period. We do not use lock-and-key mechanisms for this purpose.

Please contact Floyd Dickerson at 713-924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

Willin J. M. Comill

William McConnell

Plant Manager

cc:

Air Section Manager, TCEQ, Region 12

Director, Health and Human Services Department, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

45-3215 V8 11 6000460901



RECEIVED
US EPA, DALLAS, TX
ASSOCIATE DIRECTOR
17 JAN 27 PM 2: 59
COMPLIANCE ASSURANCE
& ENFORCEMENT DIV.

CERTIFIED MAIL: Return Receipt Requested (7011 2000 0001 4575 4942)

January 24, 2017

Houston Plant

Mr. Jeff Robinson Chief, Air Permits Section (6PD-R) U.S. Environmental Protection Agency 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

Re:

Eco Services Operations LLC Benzene NESHAP, Subpart FF, Quarterly Report

October 1, 2016 to December 31, 2016

EPA ID No.: TXD008099079

Dear Mr. Robinson:

Eco Services Operations Corp. in Houston, Texas owns and operates a Sulfuric Acid Regeneration Plant. In addition to the regeneration of sulfuric acid, the plant incinerates hazardous waste, under the conditions of the facility's RCRA Part B Permit (HW-50095).

Eco Services receives benzene waste streams from offsite customers to use as fuel in the Sulfuric Acid Regeneration Unit No. 2 (SARU) industrial furnace which is permitted under 40 CFR 266 Subpart H. Thus, the SARU industrial furnace is a treatment process for the waste and are exempt from testing and monitoring per 40 CFR 61.348(d)(1) and 61.354(a). The benzene waste streams may be stored in one or more of six treatment services (TS) storage tanks prior to treatment. The tanks are vented to the SARU industrial furnace for vapor control per 40 CFR 61.343(a). The TS Vapor Combustor provides backup vapor control for the six TS tanks. The site has no oil-water separators or individual drain systems used to convey benzene waste.

Eco Services submits this quarterly report in accordance with the reporting requirements of 40 CFR 61.357:

- Pursuant to 40 CFR 61.357(d)(6), Eco Services Operations Corp. hereby certifies that all required inspections were performed. The required inspections are itemized in Table 1.
- Pursuant to 61.357(d)(7))(iv)(G), there has been no change in the location at which the tank
 vent stream is introduced into the primary control device flame zone, the SARU industrial
 furnace.

Pursuant to 40 CFR 61.357(d)(7)(iv)(A), there have been no 3-hour periods during which the
average temperature of the gas stream in the combustion zone for the TS Vapor Combustor
was <50°F below design temperature when being used as the control device for the TS
storage tanks.

Please contact Floyd Dickerson at (713) 924-1408 if you have any comments or require any additional information on this matter.

Sincerely,

hulling 11, - Conself William McConnell

Plant Manager

cc: Air Section Manager, TCEQ, Region 12

Bureau of Air Quality Control, City of Houston

Mr. Bob Allen, Director, Harris County Pollution Control Department

Table 1

Eco Services Operations Corp. Houston, Texas Benzene Waste NESHAP Inspection Requirements For Quarterly Period Ending: <u>December 31, 2016</u>

Inspection	Was inspection performed?	Exceptions Noted
Annual Method 21 inspections of tank covers	x Yes	
and openings per 61.343(a)(1)(i)	Except as Noted	
Quarterly visual inspections of tank covers	x Yes	
and openings per 61.343(c)	Except as Noted	
Initial and annual Method 21 inspections of	x Yes	
containers per 61.345(a)(1)	Except as Noted	
Initial and quarterly visual inspections of	x Yes	
containers per 61.345(b)	Except as Noted	
Annual Method 21 inspections of treatment	x Yes	
system openings (Regeneration Unit No. 2)	Except as Noted	
per 61.348(e)(3)(ii)		
Annual Method 21 inspections of closed vent	x Yes	
systems (from tanks to TS vapor combustor	Except as Noted	
and Regeneration Unit No. 2 industrial		
furnace) per 61.349(a)(1)(i)		
Quarterly visual inspections of closed vent	x Yes	
systems and control devices (from tanks to TS	Except as Noted	
vapor combustor and Regeneration Unit No. 2		
industrial furnace, including the vapor		
combustor and Regeneration Unit No. 2		
industrial furnace) per 61.349(f)		
Daily inspections of control device continuous	x Yes	
monitoring data (temperature of TS vapor	Except as Noted	
combustor and "selected parameter" on		
Regeneration Unit No. 2 industrial furnace)		
per 61.354(c)		

Note: Where annual inspections are listed, they were not necessarily performed during this quarterly reporting period, but have been performed in the last year.

453215 V8 11 0000460901

RECEIVED US EPA, DALLAS, TX ASSOCIATE DIRECTOR

17 MAR 14 AM 4: 1 i

COMPLIANCE ASSURANCE & ENFORCEMENT DIV.

Al/Al/CO

ECOSERVICES

Eco Services Operations Corp. Houston Plant

CERTIFIED MAIL: RETURN RECEIPT REQUESTED (7015 1520 0003 4945 8170)

March 8, 2017

Mr. Jeffrey Robinson Air Permits Section Mail Code 6PD-R U.S. EPA – Region VI 1445 Ross Avenue, Suite 1200 Dallas, Texas 75202-2733

RE: Benzene Waste Operations NESHAP

Industrial Solid Waste Registration No. 31019 Hazardous Waste Permit No. HW-50095

40 CFR Part 61, Subpart FF EPA ID No. TXD008099079

Dear Mr. Robinson:

Enclosed please find a report for the 2016 calendar year Benzene Waste Operations summary for Eco Services Operations Corp.'s Houston, Texas facility. Eco Services operates a commercial industrial furnace permitted under 40 CFR Part 264 and Part 266 Subpart H by the State of Texas. This report is required under 40 CFR Part 61, Subpart FF-National Emission Standard for Benzene Waste Operations.

We have reviewed the status of each waste stream subject to regulation under this standard. In accordance with section 61.355(a), the Total Annual Benzene (TAB) quantity from this facility's waste operations was 1.9 megagrams for the operating year 2016.

Quarterly fugitive emission monitoring did not identify any emissions >500 ppm as defined in 40 CFR 61.343(a)(1)(i)(A).

Eco Services documented all daily visual inspections of the hazardous waste operations area as required in the quarterly inspection requirement as defined in 40 CFR 61.343(c). Visual inspections included sight, smell and sound observations and found no leaks in 2016.

If there are any questions, or if further information is required, please contact me at 713-924-1408.

Sincerely,

W. F. Dickerson

Environmental Consultant

xeleun

Attachment

Eco Services Operations Corp. Houston Plant 8615 Manchester Street Houston, TX 77012 CC: Air Section Manager, TCEQ, Region 12, Houston
Mr. Bob Allen, Director, Environmental Public Health Division,
Harris County Public Health and Environmental Services
City of Houston, Bureau of Air Control

Eco Services Operations Corp. Houston, Texas Calendar Year 2016 Annual Benzene Report

II (~										Mg/yr
61.357(a)(3)(vi	Annual Benzene Quantity (Mg/yr)	0 0	0.0	o c	2:0	0.0	1.9	C	0.0	1.9
61.357(a)(3)(v)	Annual Average Flow-Weighted Benzene Concentration (ppmw)	10	200	10.000	200	1,000	80,000	1,000	2,000	TOTAL
61.357(a)(3)(ii) 61.357(a)(3)(iii) 61.357(a)(3)(iv) 61.357(a)(3)(v) 61.357(a)(3)(vi) 61.357(a)(4)(4)(4)(4)(4)(4)(4)(4)(4)(4)(4)(4)(4)	Range of Benzene Concentration (ppmw)	0-10	10-200	0-10,000	0-200	0-1,000	40,000-80,000	0-1.000	10-2,000	
61.357(a)(3)(iii)	Annual Waste Quantity (Mg/yr)	0.0	9.9	0.7	1.7	0.1	23.5	1.2	0.2	
السا		Υ	Z	Z	Z	z	z	z	Υ	Y=Yes, N=No
61.357(a)(3)(i)	Water Content of Waste Stream >10%	>-	z	z	z	z	z	z	Y	Y=Yes, N= Y=Yes, N=No
7(a)(2)	Controlled Benzene Emissions	>	>	>	>	>	>	-	>	Y=Yes, N=
61.357(a)(2)	Waste Stream	9109003	9104004	0706008	1610002	1409001	1511004	0912006	9405021	-

45 3215 V8 110000 460 901



Eco Services Operations Corp. Houston Plant

September 29, 2017

Certified Mail No.: 7011 2000 0001 4575 2207

Chief, Air Branch USEPA Region 6 1445 Ross Avenue, Suite 1200 Dallas, TX 75202-2733

RE:

Eco Services Operations Corp., Houston, Texas

Title V Annual Certification Report CN: 605004464 / RN: 100220581

Permit No.: O3049

Account No.: HG-0697-O

Dear Chief, Air Branch:

Please find attached, the Annual Title V Certification Report (PCC Part 1) and Certification by Responsible Official (OP-CR01) forms for the Eco Services Houston, Texas facility which covers the period from March 3, 2017 to August 31, 2017.

If there are any questions, please contact me at (713) 924-1434 or brad.shanks@eco-services.com.

Sincerely,

Brad Shanks

Environmental Specialist

Attachments

cc:

Executive Director

Brad Shanler

Texas Commission on Environmental Quality

MC 109

P.O. Box 13087

Austin, TX 78711-3087

Air Section Manager

Texas Commission on Environmental Quality

Region 12

5425 Polk Avenue, Suite H

Houston, Texas 77023-1452

ASSOCT 0 2 2017

17 OCT -5 PM 3: 06
& ENFORCEMENT DIV

AI/AI/CO



ATTACHMENT 1

Texas Commission on Environmental Quality Federal Operating Permit Form Permit Compliance Certification - PCC (Part 1)

Texas Commission on Environmental Quality Federal Operating Permit Form Permit Compliance Certification - PCC (Part 1)	AIR CO/ HG06970	/RP
Permit Holder Name Eco Service Operations Corp	CN 605004464	
Area Name Houston Plant	O-LC-0697-O	
Operating Permit Number O 3049	Sep 29, 2017	
Certification Period Start Date [03/03/2017]	08/31/2017	
I. Certification of Continuous Compliance with Permit Terms and Conditions (Indicate response by placing a 'x' in the appropriate column for each of the following questions)	Response:	
With the possible exception of those permit terms and conditions identified in the 'Summary of Deviations' found using, at a minimum, but not limited to, the continuous or intermittent compliance method data from monitoring, recordkeeping, reporting, or testing required by the permit and any other credible evidence or information, was the permit holder in continuous compliance with all the terms and conditions of the permit over the Certification Period?	⊠ Yes	ON
II. Summary of Deviations (Indicate response by placing a 'x' in the appropriate column for each of the following questions)	Response:	
A. Were there any deviations from any permit requirements during the Certification Period that have previously been reported to the agency?		
If the answer to this question is 'Yes', please complete and attach Part 2 to this submittal.	:	:
Important Note: If previously submitted reports did not contain specific information on monitoring methods, frequency and the total number of deviations experienced over the entire certification period, then use form DevRep to provide that information.	X Yes	ON.
B. Were there any deviations from any terms or conditions of the permit during the Certification Period that are <i>currently</i> being submitted to the agency?	27/ 22	ž L

ջ ∟

Yes

 \times

If the answer to this questions is 'Yes', please include the relevant reports along with this page.

Texas Commission on Environmental Quality Federal Operating Permit Form Permit Compliance Certification - PCC (Part 2)

/RP

AIR CO/ HG06970

CN 60500464 HG-0697-0 Sep 29, 2017	(le	Report Previously Certified?	X Yes \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	T Yes No	Yes No	Yes No	Yes No	Yes [] No
CN HG	ation Period he Responsible Offici	Report Submitted To	TCEQ, EPA				The state of the s	
Permit Holder Name Eco Services Operations Corp Area Name Houston Plant Operating Permit Number 0 3049 Certification Period Start Date Beport Submittral Date Certification Period Start Date End Date	Identification of Deviation Reports Submitted During the Certification Period (Note: All reports must be certified to truth, accuracy, and completeness by the Responsible Official)	Report Description (Name of Unit, Name of Rule, Driver for Report, etc.)	Title V Deviation Report for 03/03/2017 to 08/31/2017					
Permit Holder Name Area Name Operating Permit Number Certification Period Start D	***************************************	Report Date	The state of the s	1 d a		The second secon	American control of the control of t	

TCEQ-10490 [08/12] Form PCC: This form for use by Federal Operating Permit holders and may be revised periodically.

Texas Commission on Environmental Quality Federal Operating Permit Form

/RP		
AIR CO/	CN	Description/Comments
	Date	
ermit Form s Selected (Part 3)	Customer Number Account Number Report Submittal Date	Begin End
Federal Operating Permit Form PCC - Monitoring Options Selected (Part 3)		Monitoring Option Used Citation Egamma
		SOP or GOP Index Number
		Requirement Monitored (Rule or Permit No. and Prov.)
	it Number 0	Ol due
	Area Name Area Name Operating Permit Number Certification Period Start Date	UnitiD GRPPA

TCEQ-10490 [08/12] Form PCC: This form for use by Federal Operating Permit holders and may be revised periodically.

Print Form

Page 5 of 5

Reset Form



ATTACHMENT 2

Form OP-CRO1 Certification by Responsible Official Federal Operating Permit Program



Form OP-CRO1 Certification by Responsible Official Federal Operating Permit Program

All initial permit application, revision, renewal, and reopening submittals requiring certification must be addressed using this form. Updates to site operating permit (SOP) and temporary operating permit (TOP) applications, other than public notice verification materials, must be certified prior to authorization of public notice or start of public announcement. Updates to general operating permit (GOP) applications must be certified prior to receiving an authorization to operate under a GOP.

I. IDENTIFY	ING INFORMA	TION					
RN: RN10022058	1	CN: CN60500	4464		Account No	o.: HG-06	97 - O
Permit No.: 03049)		Project No.			Supplies assessment of the supplies	<i>71</i>
Area Name: Houst	Company Name: Eco Services Operations Corp						
II. CERTIFICA	ATION TYPE (Pi	lease mark the a					
Responsible Of	ficial		Duly A	uthorized	Representa	ative	
III. SUBMITTA	L TYPE (Please	mark the appro	priate box) (Only one	response co	ın be acce _l	pted per form)
☐ SOP/TOP Initia			te to Permit				To the state of th
☐ GOP Initial Per	mit Application	Perm	it Revision, F	Renewal, o	r Reopenin	g	
Other: <u>Annual</u>	Compliance Cert	ification					
IV. CERTIFICA	TION OF TRUT	ГН					
This certification information for	reference only. Yilliam McConnell	•			n the	DAR	
(Cernj	fier Name printea	l or typed)				(RO or DA	lR)
and that, based on dated during the ti	information and l me period or on t	belief formed aft he specific date(ter reasonabl s) below, are	e inquiry, true, accu	the statemerate, and c	ents and ir omplete:	nformation
Note: Enter EITH completed. The cer	ER a Time Period	l OR Specific Da	te(s) for each	h certificat			st be
Time Period: From			to				
	St	tart Date			End Dat	e	-
Specific Dates:	09/29/17 Date 1	Date 2	Date 3	Date 4		ate5	Date 6
V	veling	Willer	w	_ Signatur	re Date: 4	1/24/	(7
Title: <u>Plant Mana</u>	ger						

4/53215 V8

110000460901



Eco Services Operations Corp. **Houston Plant**

March 27, 2018

18 APR - 2 AM 9: 20

ALLALICO

Certified Mail No.: 7015 0640 0003 9683 1335

Chief, Air Branch USEPA, Region 6 1445 Ross Avenue, Suite 1200 Dallas, TX 75202-2733

RE:

Eco Services Operations Corp (CN 605004464)

Houston Plant (RN 100220581) Title V Annual Certification Report

Permit No.: O3049

Account No.: HG-0697-O

Dear Chief, Air Branch:

Please find attached, the Annual Title V Certification Report (PCC Part 1) and Certification by Responsible Official (OP-CR01) forms for the Eco Services Houston, Texas facility which covers the period from September 1, 2017 to March 2, 2018.

If there are any questions, please contact me at (713) 924-1434 or brad.shanks@eco-services.com.

Sincerely, Brail Shanher

Brad Shanks

Environmental Specialist

Attachments

cc:

Executive Director

Texas Commission on Environmental Quality

MC 109

P.O. Box 13087

Austin, TX 78711-3087

Air Section Manager

Texas Commission on Environmental Quality

Region 12

5425 Polk Avenue, Suite H

Houston, Texas 77023-1452



Eco Services Operations Corp. Houston Plant

ATTACHMENT 1

Texas Commission on Environmental Quality
Federal Operating Permit Form
Permit Compliance Certification - PCC (Part 1 and Part 2)

Texas Commission on Environmental Quality Federal Operating Permit Form Permit Compliance Certification - PCC (Part 1)

AIR CO/ HG06970

Page 4 of 5

Texas Commission on Environmental Quality Federal Operating Permit Form Permit Compliance Certification - PCC (Part 2)

AIR CO/ HG06970

		Report Previously Certified?	X Yes No	Yes No	☐ Yes ☐ No	☐ Yes ☐ No	Yes No	Yes [No
CN 60500464 HG-0697-0 Mar 27, 2018 Mar 2, 2018	od ısible Official)	0 0 1 0						
Customer Number /CN Account Number Report Submittal Date End Date	eviation Reports Submitted During the Certification Period tified to truth, accuracy, and completeness by the Responsible Official)	Report Submitted To	TCEQ			1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		
	on Reports Submitted Du to truth, accuracy, and co	or for Report, etc.)						
Orp	Identification of Deviatic (Note: All reports must be certified t	Report Description (Name of Unit, Name of Rule, Driver for Report, etc.)	03/02/2018					
Eco Services Operations Corp Houston Plant O 3049 Sep 1, 2017	l (Note: All re	(Name of	Title V Deviation Report for 09/01/2017 to 03/02/2018					
Area Name Area Name Operating Permit Number Certification Period Start Date		Report Date	Title V Devi					

TCEQ-10490 [08/12] Form PCC: This form for use by Federal Operating Permit holders and may be revised periodically.

/RP Description/Comments Reset Form AIR CO/ 3 Report Submittal Date **Customer Number** Texas Commission on Environmental Quality Federal Operating Permit Form PCC - Monitoring Options Selected (Part 3) Account Number 띰 End Date Date Begin Specification Citation Monitoring Option Used SOP or GOP Index Number Pollutant Monitored Regulatory
Requirement
(Rule or
Permit No. 0 Group ID GRPXXXXXXX Certification Period Start Date Operating Permit Number ID Number Permit Holder Name UnitID Area Name

TCEQ-10490 [08/12] Form PCC: This form for use by Federal Operating Permit holders and may be revised periodically.

Print Form

Page 5 of 5



ATTACHMENT 2

Form OP-CRO1 Certification by Responsible Official Federal Operating Permit Program



Form OP-CRO1 Certification by Responsible Official Federal Operating Permit Program

All initial permit application, revision, renewal, and reopening submittals requiring certification must be addressed using this form. Updates to site operating permit (SOP) and temporary operating permit (TOP) applications, other than public notice verification materials, must be certified prior to authorization of public notice or start of public announcement. Updates to general operating permit (GOP) applications must be certified prior to receiving an authorization to operate under a GOP.

I. IDENTIFYING INFORMAT	TION				
RN: RN100220581	CN: CN605004	1464	Account	No.: HG-069	7 - O
Permit No.: 03049		Project No.:			
Area Name: Houston Plant		Company Na	me: Eco Services	Operations C	orp
II. CERTIFICATION TYPE (Pl	ease mark the a	ppropriate bo	x)		
Responsible Official		Duly Aut	horized Represe	ntative	
III. SUBMITTAL TYPE (Please	mark the appro	priate box) (O	nly one response	e can be accep	ted per form)
SOP/TOP Initial Permit Applicat	ion 🗌 Upda	te to Permit A _l	oplication		
☐ GOP Initial Permit Application	Perm	it Revision, Re	newal, or Reope	ning	
Other: <u>Annual Compliance Cert</u>	ification			7	
IV. CERTIFICATION OF TRUT	TH				
This certification does not exte information for reference only I,	•		s designated by that I am the		
and that, based on information and dated during the time period or on t Note: Enter EITHER a Time Period completed. The certification is not u	belief formed af he specific date d OR Specific Do	(s) below, are t ate(s) for each	rue, accurate, ar certification. Th	d complete:	
Time Period: FromS	tart Date	to	End	Date	
Specific Dates: 03/27/18 Date 1	Date 2	Date 3	Date 4	Date5	Date 6
Signature: William J Title: Plant Manager	hitor	ıll	_Signature Date	3/2	1/18

Babst Calland
Attorneys at Law

110000460901 ai/cilen

Meredith Odato Graham Attorney at Law 412-773-8712 mgraham@babstcalland.com

July 27, 2018

VIA FEDERAL EXPRESS

Chief, Environmental Enforcement
Section
Environment and Natural Resources
Division
U.S. Department of Justice
Box 7611 Ben Franklin Station
Washington, DC 20044-7611

Phillip Brooks
U.S. EPA Headquarters
William Jefferson Clinton Building
1200 Pennsylvania Avenue, N.W.
Mailcode 2242A
Washington, DC 20460

Jan Gerro
U.S. EPA Region 6
1445 Ross Avenue, Suite 1200
Mailcode 6RCEA
Dallas, TX 75202

Himanshu Vyas U.S. EPA Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6ENAT Dallas, TX 75202

Cheryl Barnett U.S. EPA Region 6 1445 Ross Avenue, Suite 1200 Mailcode 6RCEA Dallas, TX 75202

RE: DOJ No. 90-5-2-1-08500 Consent Decree Semi-Annual Report U.S. v. Rhodia Inc., USDC (N.D. Ind.), Civil Action No. 2: 07-CV-134-WCL

Ladies and Gentlemen:

In accordance with Section VII of the Consent Decree ("CD") entered in the above-entitled matter, enclosed please find the Semi-Annual Report for the Houston, Texas facility. The Report, together with the other supporting documents enclosed, satisfies the obligation to report on certain matters under the CD within 30 days after the end of each half calendar year (see CD ¶ 21-23).

If you have any questions or wish to discuss this submittal, please do not hesitate to contact me.

Sincerely,

Meredith Odato Graham

Counsel for Eco Services Operations Corp.

Enclosures



Eco Services Operations Corp. – Houston #8 and #2 Plants Consent Decree

Semi-Annual Report for Period Covering January 1, 2018 through June 30, 2018 Civil Action No.: 2: 07-CV-134-WCL

1. Effective Date:

Houston #8 - January 1, 2009 Houston #2 - April 1, 2014

2. Status of Construction or Compliance Measures Necessary to Meet Emissions Limits.

Houston #8 - The plant has now completed the construction and implementation of all compliance measures necessary to meet the Consent Decree emission limits for #8 Unit. The SO2 abatement unit was started up on November 19, 2008.

Houston #2 - Construction has been completed and implementation of all compliance measures necessary to meet the Consent Decree emission limits for #2 Unit. The SO2 abatement unit was started up on February 7, 2014.

The plant has completed the implementation of all compliance measures necessary to meet the Consent Decree emission limits.

3. <u>Compliance Issues and Proposed or Implemented Solutions</u>
Houston #8 -

Long-Term SO₂ Limit of 1.70 lbs/ton H2SO4

The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #8 unit operated below the permitted 1.70 lbs. SO₂/ton of acid produced from January 1, 2018 through June 30, 2018.

Short-Term SO₂ Limit of 3.00 lbs/ton H2SO₄

The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.



Houston #2 -

Long-Term SO₂ Limit of 1.80 lbs/ton H2SO4

The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Long-Term Limit during this reporting period. The Houston #2 unit operated below the permitted 1.80 lbs. SO₂/ton of acid produced from January 1, 2018 through June 30, 2018.

Short-Term SO₂ Limit of 3.00 lbs/ton H2SO4

The plant continuously monitored SO₂ emissions in accordance with the Consent Decree during the reporting period. At no time did the plant exceed this Short-Term Limit during this reporting period.

During the reporting period, the plant has not encountered any problems, and does not anticipate encountering any problems, with any of the conditions of the Consent Decree or any applicable permits or any other event affecting the Plant's performance under the Consent Decree.

4. <u>Status of Permit Applications</u>

Houston Title V air permit O-03049 was approved on June 28, 2012. The requirements for compliance with 40 CFR Part 60.83(a)(1) (sulfuric acid mist) Houston #2 and compliance with the Consent Decree SO2 emission rates for Houston #2 have been included as conditions in the Title V air permit.

5. Operation and Maintenance Work

The plant does not currently have any operation or maintenance work pending as a result of any of the conditions of the Consent Decree.

6. Reports to Agencies

The plant submitted the following semi-annual report (attached):

- Excess Emission Reports per 40 CFR 60.7(c)-(d) for the Houston #8 Stack SO₂/O₂ analyzers and for the Houston #2 Stack SO₂ analyzer and the Converter Inlet (Main Gas Blower) SO₂ analyzer.
- SO₂ CEMS Data Assessment Reports per 40 CFR Part 60, Appendix F, to the US Environmental Protection Agency (USEPA) and TCEQ.

ECOSERVICES

Eco Services Operations Corp. Houston Plant

Analyzer/ Pollutant/Units	Reporting Period	Accuracy Assessment
	Regen #2	
Stack SO ₂	1Q18	RATA March 12, 2018
(ppm)	2Q18	CGA May 24, 2018
Converter Inlet SO ₂	1Q18	CGA March 12, 2018
(Main Gas Blower) (ppm)	2Q18	CGA May 23, 2018
	Unit #8	
Stack SO ₂	1Q18	RATA March 14, 2018
(ppm)	2Q18	CGA May 24, 2018
	1Q18	RATA March 14, 2018
Stack O ₂	2Q18	CGA May 25, 2018

ECOSERVICES

Eco Services Operations Corp. Houston Plant

Certification Statement

I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and their attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

William McConnell

Name

Plant Manager - Houston

Title

William McConnell

Title

Onul

Signature

Plant Manager - Houston

Title



Certified Mail No.: 7015 0640 0003 9683 1526

July 13, 2018

Texas Commission on Environmental Quality Office of Permitting, Remediation and Registration Air Permits Division, MC-163 P.O. Box 13087 Austin, Texas 78711-3087

Subject:

Eco Services Operations Corp. (CN605004464)

Houston Plant (RN100220581)

Consent Decree (Civil Action No. 2:07CV134 WL) Excess Emission Report for SO2 per 40 CFR 60.7(c)-(d)

Data Assessment Report for SO2 and O2 CEMs per 40 CFR Part 60, Appendix F

Air Permit 19282 and PSD-TX-1081 Air Permit 4802 and PSD-TX-1260

Account No.: HG-0697-O

Dear Sir or Madam:

In accordance with the Consent Decree referenced above, the Eco Services Operations Corp. (Eco Services), formally Eco Services Operations LLC, Houston No. 8 became subject to 40 CFR Part 60 Subpart H, Standards of Performance for Sulfuric Acid Plants on January 1, 2009 and Houston Regen 2 became subject on April 1, 2014. Further, the Consent Decree specifies a SO2 emission standard that is more stringent than Subpart H and also incorporates an EPA-approved Alternative Monitoring Plan (AMP). As such, the semiannual excess emission report required by 40 CFR 60.7(c)-(d) and the semiannual data assessment report (DAR) required by 40 CFR Part 60 Appendix F, Procedure 1, Section 7 will address compliance with respect to the more stringent Consent Decree requirements and the Alternative Monitoring Plan. These reports are attached for the January 1, 2018 to June 30, 2018 semiannual reporting period. The relevant SO2 standards required by the Consent Decree and Alternative Monitoring Plan are as follows:

No. 8 Unit

- Per Consent Decree paragraph 11.b.i, emissions of SO2 are not to exceed a long term limit of 1.70 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per Consent Decree paragraph 11.b.ii, emissions of SO2 are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.



As discussed in the Alternative Monitoring Plan, Eco Services uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO2 and % O2) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b).

Houston Regen #2

- Per Consent Decree paragraph 11.b.viii, emissions of SO2 are not to exceed a long term limit of 1.80 pounds per ton of 100% sulfuric acid produced (averaged over all operating hours in a rolling 365-day period).
- Per Consent Decree paragraph 11.b.ii, emissions of SO2 are not to exceed a short term limit of 3.00 pounds per ton of 100% sulfuric acid produced (averaged over each rolling 3-hour period). This limit does not apply during periods of startup, shutdown, and malfunction.

As discussed in the Alternative Monitoring Plan, Eco Services uses dual analyzers to determine the conversion factor for converting monitoring data (ppm SO2) into units of the standard (lbs/ton). This exceeds the "three times daily" minimum discussed in 40 CFR 60.84(b)

Please contact me at (713) 924-1434 or <u>brad.shanks@eco-services.com</u> if you have any comments or require any additional information.

Sincerely,

Brad Shanks

Environmental Specialist

Brad Shanks

Attachments

ce: Air Section Manager, TCEQ Region 12

Mr. Bob Allen, Director, Harris County Pollution Control

Mr. Huimamshu Vyas, EPA Region 6

1445 Ross Avenue, Suite 1200, Mailcode 6ENAT, Dallas, TX 75202-2733

EPA Region 6, New Source Review Program

1445 Ross Avenue, Suite 1200, Dallas, TX 75202-2733



Certification Statement for NSPS Excess Emissions Report 40 CFR 60.7(d)

Eco Services Operations Corp. (CN605004464)
Houston, Texas Plant (RN10020581)
8615 Manchester Street
Houston, TX 77012
Air Permit 19282 and PSD-TX-1081
Air Permit 4802 and PSD-TX-1260
Account No.: HG-0697-O

Reporting Period: January 1, 2018 - June 30, 2018

I certify that the information contained in this report is true, accurate, and complete.

William McConnell
Name

Plant Manager - Houston
Title

Mullion Houston

Title



Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Summary

Reporting Period: January 1, 2018 - June 30, 2018

		Accu	racy Assessment		Any out-of-
Analyzer/ Pollutant/Units	Reporting Period	Type (RATA, CGA, or RAA)	Any Out-of- Control Periods?	Notes	control periods for Calibration Drift Assessment?**
		Rege	n #2		
	1Q18	RATA	No	 Report enclosed	No
Stack SO ₂	1010	March 12, 2018			
(ppm)	2Q18	CGA May 24, 2018	No	Report enclosed	No
		CGA	CGA		No
Converter Inlet SO ₂	1Q18	1Q18 March 12, 2018 No		Report enclosed	110
(Main Gas Blower) (ppm)	2Q18	CGA	No	Report enclosed	No
41 /	2Q16	May 23, 2018		<u> </u>	
		Unit	t # 8		
	1Q18	RATA	No	Report enclosed	No
Stack SO ₂	1010	March 14, 2018		<u> </u>	
(ppm)	2Q18	2Q18 CGA No No		Report enclosed	No
		RATA		n	No
	1Q18	March 14, 2018	No	Report enclosed	. 140
Stack O ₂	0010	CGA	No	Report enclosed	No
	2Q18	May 25, 2018	110	Troport enciosed	

Summary Report Gaseous and Opacity Excess Emission and Monitoring System Performance Regen#2 1st Semi-Annual 2018

				1
Poliutani Monitored : Re	egen #2 SO2 - Stack Analy	yzer		
Reporting Period Dates : <u>Ja</u>			June 30, 2018	
Company: <u>E</u> Address: <u>8</u>	co Services Operations Co 615 Manchester Street, Ho	orp. (CN: 60500 ouston, TX: 770	04464) Houston Plant (RN: 100220581) 012	
Process Unit(s) Description : S	ulfuric acid regeneration f	furnace with a c	caustic scrubber prior to atmospheric release	
Emission Limitation : $\frac{S}{3}$	thort-Term Emissions Lim .00 lbs SO2/Ton of 100%	it, based on the Sulfuric Acid I	permit Alternative Monitoring Plan (AMP), not to exceed produced (averaged over each rolling 3-hour period).	
<u>. I</u>	ong-Term Emissions Lim 80 lbs SO2/Ton of 100%	it, based on the Sulfuric Acid	permit Alternative Monitoring Plan (AMP), not to exceed produced (averaged over all operating hours in a rolling 365	-day period).
Monitor Manufacturer and Model:	Ametek Model 920			013619
Date of Last CMS Certification or Audit:	1Q 2018 (RATA) on:	12-Mar-18	2Q 2018 (CGA) on :	24-May-18
'Potal Time in Operating Period : -	4 185	Days Hours Hours	NSPS 40 CFR 60.7, Figure 1 requires gaseous reporting in hours	8
Total Source Operation Time :	96,3%	Uptimo		Total
<u>Reason for Excess Emission</u> Short-Term Emissions	Total Duration (Hours)		Reason for Monitor Downtime	Duration (Hours)
	N/A - limit does not apply Startup/Shutdown	y during	Monitor Equipment Malfunction:	10.0
Control Equipment Problems:	- •		Non-Monitor Equipment Malfunction:	0.0
Process Problems:			Quality Assurance Calibrations ¹ :	95.7
Other Known Causes:			Other Known Causes:	0.0
	0.0		Unknown Causes :	0.0
•			Total CMS Downtime ² :	105.7
Total Duration of Excess Emissions :			% Monitor Downtime During Total Time in Period:	2.53%
% Excess Emissions During Source Uptime : Resson for Excess Emission	Total Duration (Hours)			
Long-Term Emissions			¹ Per the AMP, the SO2 monitor is analyzed via CGAs for operating scenarios; 1. Normal operations and 2. Startuj	Statoomic and
Startup/Shutdown:		*	Maintenance periods. The monitor span values are such Normal: 0 - 500 ppm SO2	: }
Control Equipment Problems : Process Problems		•	SSM: 0 - 3,600 ppm SO2	4
		•	² The Houston plant follows the procedures specified in	the EPA approved AMP
Other Known Causes		•	for CEMS malfunctions. In accordance with the AMP,	nerally will conduct
Unknown Causes:		*	Reich testing when the stack SO2 or Converter Inlet (MCEMs malfunctions.	iain Gas Blower) 202
Total Duration of Excess Emissions		•		
% Excess Emissions During Source Uptime				
On a separate page, describe any changes si				
I certify that the information contained				1-12-18
Person Preparing Report;	Brad Shanks, Senior Er	nvironmental S	pecialist 1000 1000 1000 1000 1000 1000 1000 10	
Area Manager:	Justin Lynn	— : / /k	T(1418)	

Summary Report Gaseous and Opacity Excess Emission and Monitoring System Performance Regen#2 1st Semi-Annual 2018

Pollutant Monitored:	Regen #2 SO2 - Converter	Inlet (Main Gas	Blower) Analyzer	
Reporting Period Dates :	January 1, 2018	to	June 30, 2018	
Address:	8615 Manchester Street, H	ouston, 1X //U	4464) Houston Plant (RN: 100220581) 12	
Process Unit(s) Description :	Sulfuric acid regeneration monitor results are used (p	furnace, prior to er the AMP) for	conversion from SO2 to SO3, then to 100% sulfuric acid to the 1b SO2/ton sulfuric acid emissions calculation.	pasis. These
		diraci megguyem	nent of emissions, but used as a component to calculate fine are per the Alternate Monitoring Plan (AMP).	d emissions
Monitor Manufacturer and Model :	Ametek Model 920			
Date of Last CMS Certification or Audit:	,	12-Mar-18	2Q 2017 (CGA) on :	23-May-18
Total Time in Operating Period : Total Source Operation Time :	4,185	Days Hours Hours	NSPS 40 CFR 60.7, Figure 1 requires gaseous reporting in hours	
Total Source Operation 2000	96.3%	Uptime		Total
Reason for Excess Emission	Total Duration		A. T. Have Deposition	Duration (Hours)
Short-Term Emissions	(Hours)		Reason for Monitor Downtime	
Startup/Shutdown	:		Monitor Equipment Malfunction	
Control Equipment Problems:	Ŋ/A		Non-Monitor Equipment Malfunction:	
Process Problems	: Not a direct		Quality Assurance Calibrations ¹ :	
Other Known Causes	measurement for emissions.		Other Known Causes:	
Unknown Causes:	See above comments		Unknown Causes:	
Total Duration of Excess Emissions			Total CMS Downtime ² :	
% Excess Emissions During Source Uptime	:		% Monitor Downtime During Total Time in Period :	2.1376
<u>Reason for Excess Emission</u> <u>Long-Term Emissions</u>	Total Duration (Hours)		Per the AMP and given the high percentage (%) of SO gas stream, the monitor CGAs are spanned single range	THOU AND IN 12 VE OFF
Startup/Shutdown	1;		Additionally, only CGAs are performed each quarter of Accuracy Test Audit (RATA) is not performed.	the year, a Relative
Control Equipment Problems	11/21			
Process Problems	measurement for		² The Houston plant follows the procedures specified in	the EPA approved Al
Other Known Cause:	OIM STORM		for CEMS malfunctions. In accordance with the Alvir,	nerally will conduct
Unknown Causes	and AMP		Reich testing when the stack SO2 or Converter Inlet (M. CEMs malfunctions.	lain Gas Blower) 802
Total Duration of Excess Emission				
% Excess Emissions During Source Uptime	e: 			
On a separate page, describe any changes s				
I certify that the information contained				7-17-18
Person Preparing Report:	Brad Shanks, Senior Er	vironmental Sp	ecialist Used svew	1 16 16
Area Manager :	Justin Lynn	1	1 +112/18	

Summary Report Gaseous and Opacity Excess Emission and Monitoring System Performance Unit #8 1st Semi-Annual 2018

Pollutant Monitored : U	nit #8 O2 - Stack Analyze	<u> </u>				
Reporting Period Dates : 1						
Address: 8	npany: Eco Services Operations Corp. (CN: 605004464) Houston Plant (RN: 100220581) ddress: 8615 Manchester Street, Houston, TX 77012					
<u>n</u>	nonitor results are used to	Of the little	conversion from SO2 to SO3, then to 100% sulfuric acid to the lb SO2/ton sulfuric acid emissions calculation.	1		
		t - annurement	of emissions, but used as a component to calculate final emere per the Alternate Monitoring Plan (AMP).	issions		
Monitor Manufacturer and Model:	Ametek Model 920					
Date of Last CMS Certification or Audit:		14-Mar-18	2Q 2017 (CGA) on :	25-May-18		
Total Time in Operating Period : -	181 4,344 4,274	Days Hours Hours	NSPS 40 CFR 60:7, Figure 1 requires gaseous reporting in hours	ı		
Total Source Operation Time:	98.4%	Uptime		Total		
<u>Reason for Excess Emission</u> Short-Term Emissions	Total Duration (Hours)		Reason for Monitor Downtime	Duration (Hours)		
Startup/Shutdown:			Monitor Equipment Malfunction:	2.6		
Control Equipment Problems:	N/A		Non-Monitor Equipment Malfunction:	0.0		
Process Problems :	Not a direct		Quality Assurance Calibrations ¹ :	98.9		
Other Known Causes:	measurement for emissions.		Other Known Causes:	0.0		
Unknown Causes :	See above comments		Unknown Causes:	0.0		
Total Duration of Excess Emissions:	and AMP		Total CMS Downtime ² :	101,5		
% Excess Emissions During Source Uptime :			% Monitor Downtime During Total Time in Period ;	2.4%		
Reason for Excess Emission Long-Term Emissions	Total Duration (Hours)		Per the AMP and given the high percentage (%) of SO	2 concentration in the		
Startup/Shutdown:			gas stream, the monitor CGAs are spanned single range	ITOM U70 IU 1370 UUZ.		
Control Equipment Problems:	N/A		Accuracy Test Audit (RATA) is not performed.			
Process Problems	measurement for		² The Houston plant follows the procedures specified in	the EPA approved AMP		
Other Known Causes			for CEMS malfunctions. In accordance with the AMP,	nerally will conduct		
Unknown Causes :	and AMP		sampling with hand-held monitors when the Stack SO2 CEMS malfunction.	and O2		
Total Duration of Excess Emissions						
% Excess Emissions During Source Uptime	:					
On a separate page, describe any changes si						
I certify that the information contained				1-12-18		
Person Preparing Report:	Brad Shanks, Senior E	nvironmental S	pecialist word overhier	7/12/13		
Area Manager :	Ted Olszanski		per v			
			·			

Summary Report Gaseous and Opacity Excess Emission and Monitoring System Performance Unit #8 1st Semi-Annual 2018

			• •	1		
Pollutant Monitored: U	nit #8 SO2 - Stack Analys	zer				
Reporting Period Dates: Ja		to	June 30, 2018			
Company: <u>E</u> Address: <u>8</u>	co Services Operations C 615 Manchester Street, H	o Services Operations Corp. (CN: 605004464) Houston Plant (RN: 100220581) 15 Manchester Street, Houston, TX 77012				
			caustic scrubber prior to atmospheric release			
Emission Limitation : S	hort-Term Emissions Lin .00 lbs SO2/Ton of 100%	nit, based on the Sulfuric Acid	e permit Alternative Monitoring Plan (AMP), not to exceed produced (averaged over each rolling 3-hour period).			
<u>I</u>	ong-Term Emissions Lin .70 lbs SO2/Ton of 100%	nit, based on the Sulfuric Acid	e permit Alternative Monitoring Plan (AMP), not to exceed produced (averaged over all operating hours in a rolling 36	5-day period).		
Monitor Manufacturer and Model: 🗡	Ametek Model 920			<u>-</u>		
Date of Last CMS Certification or Audit:	1Q 2017 (RATA) on :	14-Мат-18	2Q 2017 (CGA) on :	24-May-18		
Total Time in Operating Period : -	4 274	Days Hours Hours	NSPS 40 CFR 60.7, Figure 1 requires gaseous reporting in hours			
Total Source Operation Time: -	98.4%	Uptime				
Reason for Excess Emission Short-Term Emissions	Total Duration (Hours)		Reason for Monitor Downtine	Total Duration (<u>Hours)</u>		
1 1100 F	N/A - limit does not apply	y during	Monitor Equipment Maifunction:	2.6		
Control Equipment Problems :			Non-Monitor Equipment Malfunction:	0.0		
Process Problems :			Quality Assurance Calibrations :	98.9		
Other Known Causes:	0,0		Other Known Causes:	0.0		
Unknown Causes:			Unknown Causes ;	0.0		
Total Duration of Excess Emissions:			Total CMS Downtime ²	101.5		
% Excess Emissions During Source Uptime :			% Monitor Downtime During Total Time in Period :	2.4%		
Reason for Excess Emission Long-Term Emissions	Total Duration (Hours)		¹ Per the AMP, the SO2 monitor is analyzed via CGAs f	or two different		
Startup/Shutdown:	0.0		operating scenarios; 1. Normal operations and 2. Startu- Maintenance periods. The monitor span values are such	o, Shuidown, and C		
Control Equipment Problems:	0.0		Normal: 0 - 500 ppm SO: SSM: 0 - 3,600 ppm SO:	Į.		
Process Problems:	0.0		·			
Other Known Causes;	0.0		² The Houston plant follows the procedures specified in for CEMS malfunctions. In accordance with the AMP,	during CEIMS manuncuous		
Unknown Causes:	0.0	lasting more then 24 continuous hours, Eco Services generally will con- sampling with hand-held monitors when the Stack SO2 and O2				
Total Duration of Excess Emissions :	0.0	-	CEMS malfunction.			
% Excess Emissions During Source Uptime :	0.0%	-				
On a separate page, describe any changes sin	ce last quarter in CMS, p	rocess or confro	ols: None.			
I certify that the information contained i	n this report is true, acc	urate, and com	pplete:	71716		
Person Preparing Report:	Brad Shanks, Senior Er		1 \ 44 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	7-12-18		
Area Manager :	Ted Olszanski		Jell Marysh	6/11/18_		

ECOSERVICES

Eco Services Operations Corp. Houston Plant

Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Regen #2

Stack SO₂ Analyzer

1st Quarter 2018 - RATA

RELATIVE ACCURACY TEST AUDIT REPORT ECO SERVICES OPERATIONS CORP. SULFURIC ACID REGENERATION UNIT NO. 2 HOUSTON, TEXAS TEST DATE: 12 MARCH 2018 RN 100220581/CN 605004464 PERMIT NO. 4802

Prepared for:

ECO SERVICES OPERATIONS CORP.

8715 Manchester Street Houston, TX 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way West Chester, Pennsylvania 19380

April 2018

W.O. No. 15364.002.006

1. TEST PROGRAM SUMMARY

1.1 INTRODUCTION

The Eco Services Operations Corp. (Eco Services) Houston, TX facility is required to perform annual continuous emissions monitoring system (CEMS) Relative Accuracy Test Audits (RATAs) on its Sulfuric Acid Regeneration Unit No. 2 (Regen 2) scrubber stack dual range CEMS and the main gas blower CEMS. This report provides a detailed description of the monitoring procedures that were used to demonstrate compliance for each sulfur dioxide (SO₂) CEMS.

Section 1 provides a summary of the overall test program and test parameters. Section 2 provides a summary of the Relative Accuracy Test Audit (RATA) results for each CEMS tested. A description of process operations, test locations, and sampling procedures are provided in Sections 3 through 5, respectively.

Appendices A through E provide Regen 2 CEMS and process operations data, reference method CEMS data, example calculations, quality control data, and project participants, respectively.

1.2 SAMPLE PROGRAM MATRIX

Table 1-1 provides a summary of test parameters and test procedures. All CEMS RATA testing was performed on 12 and 14 March 2018.

Table 1-1
Sample Program Matrix

Sample Parameter	Sample Location	Test Method	Number of Tests	Comments
Sulfur Dioxide	Stack CEMS	EPA Method 6C	9	ppmv and % relative accuracy
Sulfur Dioxide	Main Gas Blower CEMS	Performance Specification 2 Alternative Method Section 16.2	2 x 3 ¹	% SO ₂ and % difference

^{1.} Two reference gas cylinders were used, each cylinder challenged the SO₂ analyzer with the known SO₂ concentration three separate times.

2. RESULTS AND DISCUSSION

The CEMS certification results are shown in Table 2-1. The test results for relative accuracy met the performance specification criteria for each CEMS tested. The Eco Services stack SO₂ CEMS operates at two ranges, 0-500 ppm and 0-3,600 ppm. One RATA test was used to evaluate both CEMS ranges. Nine reference method test runs were conducted at the stack CEMS location. The relative accuracy was based upon all nine runs.

Appendix B provides detailed summaries of the relative accuracy and performance specification testing.

Table 2-1
Summary of CEMS Performance Specification Test Results
RN 100220581/CN 605004464, Permit No. 4802

	Relative Accuracy				
Stack Analyzers	Performance Required (%)	Performance Demonstrated (%)			
SO ₂	20	13.5			

Converter		e Accuracy e Method 16.2)
Analyzer (Main Gas Blower)	Performance Required (%)	Performance Demonstrated (%)
SO_2	15	1.7 (low conc.) 1.0 (mid conc.)

3. DESCRIPTION OF PROCESS OPERATIONS

3.1 DESCRIPTION OF SULFURIC ACID REGENERATION UNIT NO. 2

Eco Services typically injects and atomizes spent sulfuric acids, raw materials, and/or fuels into a decomposition furnace operating at temperatures between 1884°F and 2127°F, with a retention time of approximately three seconds. After decomposition, the exhaust gas passes through a waste heat boiler then enters the gas purification system. Sulfuric Acid Regeneration Unit No. 2 contains a gas purification system consisting of the following:

- · A quench tower.
- A wet gas cooler.
- Two (2) wet electrostatic precipitators (ESP).

The gas purification equipment up to and including the wet ESPs is considered part of the RCRA operations. The sulfuric acid production process begins at the gas drying tower and consists of the following:

- A gas drying tower with mist filters.
- A SO₂ to SO₃ converter.
- An oleum tower.
- A SO₃ absorber.
- A mist eliminator.
- A SO₂ scrubber

Dilute sulfuric acid from the process is reused in the sulfuric acid production process, or neutralized and discharged subject to permits and provisions of local, state, and federal regulations.

4. SULFUR DIOXIDE CONTINUOUS EMISSIONS MONITORS (CEMS), LOCATIONS AND SPECIFICATIONS

Eco Services operates CEMS to measure SO_2 concentrations at the stack and the main gas . blower/converter inlet. The SO_2 CEMS specifications are:

Location Manufacturer		Serial Number	Range		
			Dual range:		
Stack	Ametek	ZY-920-10628-1	Low: 0-500 ppm SO ₂		
			High: 0-3,600 ppm SO ₂		
Main Gas Blower	Ametek	AW-920-9965-1	Single range: 0-15% SO ₂		



Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Regen #2

Stack SO₂ Analyzer

2nd Quarter 2018 - CGA



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Regen 2 Stack SO₂ Analyzer (Exit)

Low Range

Date: 5/24/2018	9:39AM	,	n; <u>J. Thor</u>	mas \			
Serial Number: ZY	Si	gnature: 📐					
Cylinder ID number	CC456812		CC146576				
Date of Certification	5/1/2018		4/30/2018				
Type of certification (e.g. EPA Protocol 1 or CRM).	EPA Pro.	1 .	EPA Pro.	1			
	Trial 1		; Tr	ial 2	Trial 3		
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	
Certified audit value C_a (ppm)	125,4	277	125,4	277	125.4	277	
CEM Response value C_m (ppm)	126	277	126	277	126	277	
Accuracy A (% or ppm)	0.478%	0%	0.478%	0%	0.478%	0%	

where $A = (C_m - C_n) \times 100$ C_n an Air Liquide company

Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS Grade of Product: EPA Protocol

Customer:

ECO SERVICES

Part Number:

E02NI99E15A0005

Cylinder Number:

CC456812

Laboratory: PGVP Number: 124 - Pasadena (SG06) - TX A32018

Gas Code:

SO2, BALN

Reference Number:

163-401184011-1

Cylinder Volume:

144.4 CF 2015 PSIG

Cylinder Pressure: Valve Outlet:

660

Certification Date:

May 01, 2018

Expiration Date: May 01, 2026

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical source talents are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

e This Cylinder below 100 psig, i.e. 0.7 megapascals.

			Do Not Use This Cylinder Di	Slow 100 bard' iro:	o., incgapas	NAME OF TAXABLE PARTY.			
(413-11-2-1-2-1-2-1-2-1-2-1-2-1-2-1-2-1-2-	ANALYTICAL RESULTS								
Component Requested Concentration		Actual Concentration	Protocol Method	Total R Uncert		Assay Dates			
SULFUR DIG		125.0 PPM	125.4 PPM	G1	+/- 1.0%	+/- 1.0% NIST Traceable 04/24/201			
NITROGEN		Balance			-				
CALIBRATION STANDARDS									
Туре	Lot ID	Cylinder No	Concentration			Uncertainty	Expiration Date		
NTRM	150606	CC449879	248.1 PPM SULF			+/-0.60% +/-0.8%	Dec 17, 2020 Jul 28, 2023		
NTRM	11010413		99.6 PPM SULFU	IR DIOXIDE/NI	ROGEN	+7-U.B76	30120, 2020		
ANALYTICAL EQUIPMENT									
l Instrumen	t/Make/Mc	del	Analytical Principle		Last Multipoint C	Calibration			
<u> </u>		AHR0600411	FTIR	no e ga halandang panag		Apr 12, 2018			

Triad Data Available Upon Request





Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS **Grade of Product: EPA Protocol**

Customer:

ECO SERVICES

STOREROOM

Part Number:

E02NI99E15A3823

Cylinder Number:

CC146576

Laboratory: PGVP Number: 124 - Pasadena (SG06) - TX

Gas Code:

A32018

SO2,BALN

Reference Number: 163-401184005-1

Cylinder Volume:

144.4 Cubic Feet

Cylinder Pressure:

2015 PSIG

Valve Outlet:

660

Certification Date:

Apr 30, 2018

Expiration Date: Apr 30, 2026

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (way 2012) document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

жылун этрэлэг альаў ў фана Мат	and the same of the same of the same	ing padagan sagaran pada na pada na sana na pada	ANALYTI	CAL RESU	LTS			
		Requested Concentration	Actual Concentration			elative Inty	Assay Dates	
SULFUR DIOXIDE 275.0 PPM		277.0 PPM	G1	+/- 1.1% NIST Traceable		04/23/2018, 04/30/2018		
NITROGE	١ .	Balance						
Туре	Lot ID	Cylinder No	CALIBRATI Concentration	ON STANI	ARDS	Uncertainty	Expiration Date	
NTRM	150606	CC450512	248.1 PPM SULFU	JR DIOXIDE/NIT	ROGEN	+/-0.60%	Dec 17, 2020	
			ANALYTIC	AL EOUIP	MENT			
Instrument/Make/Model		Analytical Principle			Last Multipoint Galibration			
SO2-L - (2) NICOLET 6700 AHR0600412		FTIR			Apr 25, 2018			

Triad Data Available Upon Request





Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist Regen 2 Stack SO₂ Analyzer (Exit)

High Range

Technician: J. Thomas Time: 9:39AM Date: 5/24/2018 Signature: ZY-920-10628-1 Serial Number: CC98663. Cylinder ID number CC400268 4/30/2018 5/1/2018 Date of Certification EPA Pro, 1 EPA Pro. 1 Type of certification (e.g. EPA Protocol 1 or CRM). Trial 3 Trial 2 Trial 1 Audit Audit Audit Audit Audit Audit Point 2 Point 1 Point 2 Point,1 Point 2 Point 1 1986 899.6 899.6 1986 1986 899,6 Certified audit value Ca (ppm) 2002 905 2001 904 1998 902 CEM Response value C_m (ppm) 0.806% 0.600% 0.755% 0.489% Accuracy A (% or ppm) 0.604% 0.267%

where $A = (C_m - C_n) \times 100$ C_n



Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS **Grade of Product: EPA Protocol**

Customer:

ECO SERVICES

STOREROOM

Part Number:

E02NI99E15A0051

Cylinder Number:

CC400268

Laboratory: PGVP Number: 124 - Pasadena (SG06) - TX

Gas Code:

A32018 SO2,BALN

Reference Number:

163-401184185-1

Cylinder Volume:

144.4 CF 2015 PSIG

Cylinder Pressure: Valve Outlet:

660

Certification Date:

May 01, 2018

Expiration Date: May 01, 2026

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

	The hope of the state of the st	to seem to provide the control of th	DO MOLOSE TRIS CARIDOL I	and the second of the second o	an elektrich mit der gelegen der gelegen gestellt gegeben der gelegen der gegeben der gelegen der gegeben der g			
Compone	ent	Requested	ANALYTI Actual	CAL RESU Protocol	Total Rela		Assay	
		Concentration	Concentration	ncentration Method		ty	Dates	
SULFUR D	OOXIDE	900.0 PPM	899,6 PPM	G1	+/- 0,7% NI	ST Traceable	04/24/2018, 05/01/2018	
NITROGE		Balance			-			
			CALIBRATI	CANT COLVEN	MDDC	Will the state of	NA THE STATE OF TH	
Туре	Lot ID	Cylinder No	Concentration		ARDS	Uncertainty	Expiration Date	
NTRM	11010326	KAL004765	968.8 PPM SULF	UR DIOXIDE/NI	TROGEN	+/-0.6%	May 30, 2023	
Instrume	nt/Make/Mo	odel	ANALYTIC Analytic	AL EQUIP	MENT	Last Multipoint (Calibration	
SO2-M - (2) NICOLET 6700 AHR0600412		FTIR .			Арг 25, 2018			

Triad Data Available Upon Request





Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS

Grade of Product: EPA Protocol

Customer.

ECO SERVICES

STOREROOM

Part Number:

E02NI99E15A0472

Cylinder Number:

CC98663 124 - Pasadena (SG06) - TX

Laboratory: PGVP Number:

A32018

Gas Code:

SO2,BALN

Reference Number:

163-401184181-1

Cylinder Volume:

144.5 CF 2015 PSIG

Cylinder Pressure: Valve Outlet:

660

Certification Date:

Apr 30, 2018

Expiration Date: Apr 30, 2026

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Celibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical Interference. This cylinder has a total analytical 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

reconstitution constitution is the	anna kampini ya kanpun kanca 1949 h.	THE TO SEE THE PROPERTY OF THE	ANALYTI	CAL RESU	LTS		
Component Requested Concentration		Actual Concentration	Protocol Method	Total Relative Uncertainty		Assay Dates	
SULFUR DIOXIDE 1980 PPM		1986 PPM	G1	+/- 0.7% NIST Traceable		04/23/2018, 04/30/2018	
NITROGEN Balance					enganining same		
Type	Lot ID	Cylinder No	CALIBRATIC Concentration	ON STANI	DARDS	Uncertainty	Expiration Date
Type	120105	KAL003365	2502 PPM SULFU	R DIOXIDE/NIT	ROGEN	+/-0.60%	Jun 04, 2019
	nt/Make/M	ndel	ANALYTIC Analytic	AL EQUIP	MENT	Last Multipoint	Calibration .
Instrument/Make/Model SO2-H - (2) NICOLET 6700 AHR0600412			FTIR		Apr 25, 2018		

Triad Data Available Upon Request





Data Assessment Reports
40 CFR Part 60 Appendix F
Procedure 1, Section 7

Regen #2

Converter Inlet (Main Gas Blower) SO2 Analyzer

1st Quarter 2018 - CGA

RELATIVE ACCURACY TEST AUDIT REPORT ECO SERVICES OPERATIONS CORP. SULFURIC ACID REGENERATION UNIT NO. 2 HOUSTON, TEXAS TEST DATE: 12 MARCH 2018 RN 100220581/CN 605004464 PERMIT NO. 4802

Prepared for:

ECO SERVICES OPERATIONS CORP.

8715 Manchester Street Houston, TX 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way West Chester, Pennsylvania 19380

April 2018

W.O. No. 15364.002.006

1. TEST PROGRAM SUMMARY

1.1 INTRODUCTION

The Eco Services Operations Corp. (Eco Services) Houston, TX facility is required to perform annual continuous emissions monitoring system (CEMS) Relative Accuracy Test Audits (RATAs) on its Sulfuric Acid Regeneration Unit No. 2 (Regen 2) scrubber stack dual range CEMS and the main gas blower CEMS. This report provides a detailed description of the monitoring procedures that were used to demonstrate compliance for each sulfur dioxide (SO₂) CEMS.

Section 1 provides a summary of the overall test program and test parameters. Section 2 provides a summary of the Relative Accuracy Test Audit (RATA) results for each CEMS tested. A description of process operations, test locations, and sampling procedures are provided in Sections 3 through 5, respectively.

Appendices A through E provide Regen 2 CEMS and process operations data, reference method CEMS data, example calculations, quality control data, and project participants, respectively.

1.2 SAMPLE PROGRAM MATRIX

Table 1-1 provides a summary of test parameters and test procedures. All CEMS RATA testing was performed on 12 and 14 March 2018.

Table 1-1
Sample Program Matrix

Sample Parameter	Sample Location	Test Method	Number of Tests	Comments
Sulfur Dioxide	Stack CEMS	EPA Method 6C	9	ppmv and % relative accuracy
Sulfur Dioxide	Main Gas Blower CEMS	Performance Specification 2 Alternative Method Section 16.2	2 x 3 ¹	% SO ₂ and % difference

^{1.} Two reference gas cylinders were used, each cylinder challenged the SO₂ analyzer with the known SO₂ concentration three separate times.

2. RESULTS AND DISCUSSION

The CEMS certification results are shown in Table 2-1. The test results for relative accuracy met the performance specification criteria for each CEMS tested. The Eco Services stack SO₂ CEMS operates at two ranges, 0-500 ppm and 0-3,600 ppm. One RATA test was used to evaluate both CEMS ranges. Nine reference method test runs were conducted at the stack CEMS location. The relative accuracy was based upon all nine runs.

Appendix B provides detailed summaries of the relative accuracy and performance specification testing.

Table 2-1
Summary of CEMS Performance Specification Test Results
RN 100220581/CN 605004464, Permit No. 4802

	Relative Accuracy		
Stack Analyzers	Performance Required (%)	Performance Demonstrated (%)	
SO_2	20	13.5	

Converter	Relative Accuracy (Alternative Method 16.2)		
Analyzer (Main Gas Blower)	Performance Required (%)	Performance Demonstrated (%)	
SO_2	15	1.7 (low conc.) 1.0 (mid conc.)	

3. DESCRIPTION OF PROCESS OPERATIONS

3.1 DESCRIPTION OF SULFURIC ACID REGENERATION UNIT NO. 2

Eco Services typically injects and atomizes spent sulfuric acids, raw materials, and/or fuels into a decomposition furnace operating at temperatures between 1884°F and 2127°F, with a retention time of approximately three seconds. After decomposition, the exhaust gas passes through a waste heat boiler then enters the gas purification system. Sulfuric Acid Regeneration Unit No. 2 contains a gas purification system consisting of the following:

- A quench tower.
- A wet gas cooler.
- Two (2) wet electrostatic precipitators (ESP).

The gas purification equipment up to and including the wet ESPs is considered part of the RCRA operations. The sulfuric acid production process begins at the gas drying tower and consists of the following:

- A gas drying tower with mist filters.
- A SO₂ to SO₃ converter.
- An oleum tower.
- A SO₃ absorber.
- A mist eliminator.
- A SO₂ scrubber

Dilute sulfuric acid from the process is reused in the sulfuric acid production process, or neutralized and discharged subject to permits and provisions of local, state, and federal regulations.

4. SULFUR DIOXIDE CONTINUOUS EMISSIONS MONITORS (CEMS), LOCATIONS AND SPECIFICATIONS

Eco Services operates CEMS to measure SO₂ concentrations at the stack and the main gas blower/converter inlet. The SO₂ CEMS specifications are:

Location	Manufacturer	Serial Number	Range
Stack			Dual range:
	Ametek	ZY-920-10628-1	Low: 0-500 ppm SO ₂
			High: 0-3,600 ppm SO ₂
Main Gas Blower	Ametek	AW-920-9965-1	Single range: 0-15% SO ₂

6



Eco Services Operations Corp. Houston Plant

<u>Data Assessment Reports</u> 40 CFR Part 60 Appendix F <u>Procedure 1, Section 7</u>

Regen #2

Converter Inlet (Main Gas Blower) SO₂ Analyzer

2nd Quarter 2018 - CGA



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist MGB SO₂ Analyzer

Signature:

Date: <u>5/23/2018</u> Tin Serial Number: AW-920-9965-1

Time: 11:18AM

Technician: Jeremy Thomas

CC505202 SG9112097 Cylinder ID number Date of Certification 5/1/2018 11/29/2016 EPA Pro, 1 Type of certification EPA Pro. 1 (e.g. EPA Protocol 1 or CRM). Trial 1 Trial 2 Trial 3 Audit Audit Audit Audit Audit Audit Point 1 Point 2 Point 1 Point 2 Point 2 Point 1 8,187 3,956 8.187 Certified audit value C_a 3.956 8.187 3.956, (ppm) CEM Response value 3.95 8.16 3.95 8.17 3,94 8.15 C_m (ppm) Accuracy A (% or ppm) -0152% -0.152% -0.208% -0.404% -0.452% -0.330%

where $A = (C_m - C_a) \times 100$ C_a



Airgas Specialty Gases Airgas USA, LLC 630 United Drive Durham, NC 27713 Airgas.com

CERTIFICATE OF ANALYSIS

Grade of Product: EPA Protocol

Part Number: Cylinder Number: E04Ni81E15A25V4

SG9112097

124 - Durham (SAP) - NC

Gas Code:

Laboratory:

PGVP Number:

B22014

CO,NO,NOX,SO2,BALN

Reference Number: 122-124446588-1

Cylinder Volume: Cylinder Pressure:

155.4 CF 2015 PSIG

Valve Outlet:

660

Certification Date:

Aug 11, 2014

Expiration Date: Aug 11, 2022

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, I.e. 0.7 megapascals

ANALYTICAL RESULTS						
Component	Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty	Assay Dates	
NOX	270.0 PPM	272.7 PPM	G1	+/- 0,8% NIST Traceable	08/04/2014, 08/11/2014	
NITRIC OXIDE	270.0 PPM	272,7 PPM	G1	+/- 0.8% NIST Traceable	08/04/2014, 08/11/2014	
SULFUR DIOXIDE	450.0 PPM	462.9 PPM	G1	+/- 0.9% NIST Traceable	08/04/2014, 08/11/2014	
CARBON DIOXIDE	18.00 %	17.92 %	G1	+/- 0.7% NIST Traceable	08/04/2014	
NITROGEN	Balance			**		

Type	Lot ID	Cylinder No	CALIBRATION STANDARDS Concentration	Uncertainty	Expiration Date
NTRM	12061908	CC367439	250.8 PPM NITRIC OXIDE/NITROGEN	+/- 0,5%	May 04, 2018
PRM	12329	726612	25.02 PPM NITROGEN DIOXIDE/NITROGEN	+/- 1.5%	Feb 14, 2012
GMIS	0207201403	CC500952	14.95 PPM NITROGEN DIOXIDE/NITROGEN	+/- 1.6%	Feb 07, 2017
NTRM	12062928	CC407422	483.1 PPM SULFUR DIOXIDE/NITROGEN	+/- 0,6%	Jul 18, 2018
NTRM	12062526	CC354757	19.87 % CARBON DIOXIDE/NITROGEN	+/- 0,6%	Jan 27, 2018
NIKW The SRM			to the GMIS used in the assay and not part of the analysis.		

	ANALYTICAL EQUIPA	
Instrument/Make/Model	Analytical Principle	Last Multipoint Calibration
Nicolet 6700 AHR0801549 CO2	FTIR	Jul 31, 2014
Nicolet 6700 AHR0801549 NO	FTIR .	.jul 31, 2014
Nicolet 6700 AHR0801549 NO	FTIR	Jul 31, 2014
Nicolet 6700 AHR0801549 SO2	FTIR	Jul 31, 2014

Triad Data Available Upon Request





Airgas Specialty Gases Airgas USA, LLC 6141 Easton Road Bldg 1 Plumsteadville, PA 18949 Airgas.com

CERTIFICATE OF ANALYSIS Grade of Product: CERTIFIED STANDARD-SPEC

Part Number:

X02NI91C15A0012

Cylinder Number:

Laboratory:

Analysis Ďate: Lot Number:

CC505202 124 - Plumsteadville - PA

May 01, 2018

160-401184019-1 Expiration Date: May 01, 2026

Reference Number:

Cylinder Volume: Cylinder Pressure:

Valve Outlet:

160-401184019-1

30.7 CF 407 PSIG

660

Product composition verified by direct comparison to calibration standards traceable to N.I.S.T. weights and/or N.I.S.T. Gas Mixture reference materials.

- and the contract of the cont	A A	ALYTICAL RESULTS	•
Component	Reg Conc	Actual Concentration	Analytical
Component	·	(Volume %)	Uncertainty
THE DIOVIDE	8.260 %	8.187 %	+/- 2%
SULFUR DIOXIDE NITROGEN	Balance	Balance	





Eco Services Operations Corp. Houston Plant

Data Assessment Reports
40 CFR Part 60 Appendix F
Procedure 1, Section 7

Unit #8

Stack SO₂ Analyzer

1st Quarter 2018 – RATA

RELATIVE ACCURACY TEST AUDIT REPORT ECO SERVICES OPERATIONS CORP. SULFURIC ACID UNIT NO. 8 HOUSTON, TEXAS TEST DATE: 14 MARCH 2018 RN 100220581/CN 605004464 PERMIT NO. 19282

Prepared for:

ECO SERVICES OPERATIONS CORP.

8715 Manchester Street Houston, TX 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way West Chester, Pennsylvania 19380

April 2018

W.O. No. 15364.002.006

1. TEST PROGRAM SUMMARY

1.1 INTRODUCTION

The Eco Services Operations Corp. (Eco Services) Houston, TX facility is required to perform annual continuous emissions monitoring system (CEMS) Relative Accuracy Test Audits (RATAs) on its Sulfuric Acid Unit No. 8 (Unit 8) scrubber stack. This report provides a detailed description of the monitoring procedures that were used to demonstrate compliance for the sulfur dioxide (SO₂) and oxygen (O₂) CEMS.

Section 1 provides a summary of the overall test program and test parameters. Section 2 provides a summary of the Relative Accuracy Test Audit (RATA) results for each CEMS tested. A description of process operations, test locations, and sampling procedures are provided in Sections 3 through 5, respectively.

Appendices A through E provide Unit 8 CEMS and process operations data, reference method CEMS data, example calculations, quality control data, and project participants, respectively.

1.2 SAMPLE PROGRAM MATRIX

Table 1-1 provides a summary of test parameters and test procedures. All CEMS RATA testing was performed on 14 March 2018.

Table 1-1
Sample Program Matrix

	Comments	Number of Tests	Test Method	Sample Location	Sample
relative	nnmy and % related			11000000	Parameter
	accuracy	11	EPA Method 6C	Stack CEMS	Sulfur Dioxide
1 %	% O ₂ and %				
ce	difference	11	EPA Method 3A	Stack CEMS	Oxygen
у 1 %	% O2 and %	11		Stack CEMS	

2. RESULTS AND DISCUSSION

The CEMS certification results are shown in Table 2-1. The test results for relative accuracy met the performance specification criteria for each CEMS tested. Eleven reference method test runs were conducted at the stack CEMS location. The relative accuracy was based upon the best nine of the eleven runs. Run No. 1 was not used in the RATA calculations due to a gas conditioner failure in the Weston CEM system. All eleven RATA test runs are reported in the appendices.

Appendix B provides detailed summaries of the relative accuracy and performance specification testing.

Table 2-1
Summary of CEMS Performance Specification Test Results
RN 100220581/CN 605004464, Permit No. 19282

	Relative Accuracy		
Stack Analyzers	Performance Required (%)	Performance Demonstrated (%)	
SO ₂	≤ 20	4.7	
O ₂	≤1	0.01	

3. DESCRIPTION OF PROCESS OPERATIONS

3.1 DESCRIPTION OF SULFURIC ACID UNIT NO. 8

The Eco Services Sulfuric Acid Unit No. 8 is a virgin sulfuric acid manufacturing unit. In the virgin sulfuric acid process, molten sulfur is sprayed into the furnace and burned at a high temperature with excess air to produce sulfur dioxide (SO₂); the SO₂ is cooled in a waste heat boiler. Gaseous SO₂ is reacted with air in a catalytic converter to produce sulfur trioxide (SO₃); between the first two stages of the converter, exothermically generated heat is removed by another waste heat boiler. The SO₃ is absorbed in strong acid in an absorbing tower to produce high-concentration sulfuric acid. Exhaust gas leaves the absorbing tower passing through an SO₂ scrubber before exiting from the stack.

4. SULFUR DIOXIDE CONTINUOUS EMISSIONS MONITORS (CEMS), LOCATIONS AND SPECIFICATIONS

Eco Services operates CEMS to measure SO₂ and O₂ concentrations at the scrubber stack. The CEMS specifications are:

Location	Manufacturer	Serial Number	Range
	2 000	VE-920-8700-2	0-5000 ppm SO ₂
Stack SO ₂		VE-920-8700-2	0-25% O₂
Stack O2	Ametek 920		



Eco Services Operations Corp. Houston Plant

Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Unit #8

Stack SO₂ Analyzer

2nd Quarter 2018 - CGA



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist U8 Stack SO2 Analyzer

Date: 5/25/2018

Time: 9:00AM

Technician: J. Thomas

Serial Number: VE-920-8700-2

Signature:

Low Range

			1	<u> </u>	1	
Cylinder ID number	CC456812		CC146576	CC146576		
Date of Certification	5/1/2018	5/1/2018		4/30/2018		
Type of certification (e.g. EPA Protocol 1 or CRM).	EPA Pro. 1 Trial 1		EPA Pro.	1		
			Tri	al 2	Trial 3	
	Audit Point 1	Audit Point 2	Audit Point l	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a (ppm)	125.4	277	125.4	277	125.4	277
CEM Response value C_m (ppm)	124	278	125	279	125	278
Accuracy A (% or ppm)	-1.116%	0.361%	-0.319%	0.722%	-0.319%	0.361%

where $A = (C_m - C_a) \times 100$ C_a



Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS **Grade of Product: EPA Protocol**

Customer:

ECO SERVICES

Part Number:

E02NI99E15A0005

Cylinder Number:

CC456812

Laboratory: PGVP Number: 124 - Pasadena (SG06) - TX

A32018

SO2,BALN Gas Code:

163-401184011-1 Reference Number:

Cylinder Volume:

144.4 CF 2015 PSIG

Cylinder Pressure: Valve Outlet:

660

Certification Date:

May 01, 2018

Expiration Date: May 01, 2026

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

CANADO NO SERVICIO DE CANADO	ngovennosta en la composito de la composito de	and the same of	DO HOLOSO 1593 CAUTEC	Co-Managarahagaranan da managaran da managar	ROMANIA IN COLUMN SANAVOLI	VALIDAJII II 1900 INI INI TANGENI WALIONI WALI	September 19 Septe	
ANALYTICAL RESULTS								
		Requested Concentration	Actual Concentration	Protocol Total R tion Method Uncert			Assay Dates	
SULFUR DIOXIDE 125.0 PPM		125,4 PPM	G1	+/- 1.0%	NIST Traceable	04/24/2018, 05/01/2018		
NITROGEN		Bajance						
CALIBRATION STANDARDS								
Туре	Lot ID	Cylinder No	Concentration			Uncertainty	Expiration Date	
NTRM	150606	CC449879		FUR DIOXIDE/NI		+/-0,60%	Dec 17, 2020	
NTRM	11010413	KAL004791	99,6 PPM SULF	UR DIOXIDE/NIT	ROGEN	+/-0,8%	Jul 28, 2023	
ANALYTICAL EQUIPMENT Instrument/Make/Model Analytical Principle Last Multipoint Calibration						alibration		
SO2-M - NICOLET 6700 AHR0600411			FIIR			Арг 12, 2018		

Triad Data Available Upon Request





Airgas Specialty Gases Airgas USA, LLC 9810 BAY AREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS **Grade of Product: EPA Protocol**

Customer:

ECO SERVICES

STOREROOM

Part Number:

E02NI99E15A3823

Cylinder Number: Laboratory:

CC146576 124 - Pasadena (SG06) - TX

PGVP Number:

A32018

Gas Code:

SO2, BALN

Expiration Date: Apr 30, 2026

Reference Number: Cylinder Volume:

163-401184005-1 144.4 Cubic Feet

2015 PSIG Cylinder Pressure:

660 Valve Outlet: Арг 30, 2018 Certification Date:

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a uncertainty as the confidence of the use of the us

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

ganga di kasansa palami sani pad	<u>Linguis in institution (in including </u>	Marie (Marie	ANALYT	ICAL RESU	LTS		Assay
Component SULFUR DIOXIDE		Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty		Dates
		275.0 PPM	277.0 PPM	G1	+/- 1.1%	NIST Traceable	04/23/2018, 04/30/2018
NITROGEN		Balance					
T	Lot ID	Cylinder No	CALIBRAT Concentration			Uncertainty	Expiration Date
Type NTRM	150606	CC450512	248.1 PPM SULF	UR DIOXIDE/NIT	ROGEN	+/-0.60%	Dec 17, 2020
·			ANALYTICAL EQUIPMENT Analytical Principle		MENT	Last Multipoint	Calibration
Instrument/Make/Model SO2-L - (2) NICOLET 6700 AHR0600412			FTIR		Apr 25, 2018		

Triad Data Available Upon Request





Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist U8 Stack SO2 Analyzer

Date: 5/25/2018

Time: 9:00AM

Technician: J. Thomas Signature:

Revised: 05/28/18

Serial Number: VE-920-8700-2

High Range

High Range			1			
Cylinder ID number	ALM04152	26	ALM00486	8		
Date of Certification	1/16/2012 EPA Pro. 1		12/21/2016			
Type of certification (e.g. EPA Protocol 1 or CRM).			EPA Pro. 1			
Civiat).	Trial 1		Trial 2		Trial 3	
	Audit Point 1	Audit Point 2	Audit Point l	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value Ca	909	2001	909	2001	909	2001
(ppm) CEM Response value	889	1979	904	1991	907	1996
C _m (ppm) Accuracy A (% or ppm)	-2.200%	-1.099%	-0.550%	0.500%	-0.220%	0250%

where $A = (C_m - C_n) \times 100$ C_n



Airgas Specialty Gases Airgas USA, LLC 9810 BAY ÁREA BLVD Pasadena, TX 77507 Airgas.com

CERTIFICATE OF ANALYSIS

Grade of Product: CERTIFIED STANDARD-SPEC

Customer:, Part Number:

Cylinder Number: Laboratory:

Analysis Ďate:

Lot Number:

SOLVAY USA INC - STOREROOM

X02NI99C15A06L5 ALM041526

124 - Pasadena (SG06) - TX

Jan 16, 2017 163-400829222-1

Reference Number: Cylinder Volume: Cylinder Pressure: Valve Outlet:

163-400829222-1

144,4 CF 2015 PSIG 660

Expiration Date: , Jan 16, 2020

Product composition verified by direct comparison to calibration standards traceable to N.I.S.T. weights and/or N.I.S.T.

Gas Mixture reference materials.

ANALYTICAL RESULTS

Component

Req Conc

Actual Concentration

(Mole %)

Analytical Uncertainty

SULFUR DIOXIDE NITROGEN

906.0 PPM Balance

909,0 PPM

+/- 2%





Airgas USA, LLC 9810 BAY ARBA BLVD Pasadena, TX 77507 Alrgas.com

CERTIFICATE OF ANALYSIS Grade of Product: EPA Protocol

Part Number: Cylinder Number: E02NI99E15A01H5

ALM004868

124 - Pasadena (SG06) - TX

Laboratory: PGVP Number: Gas Code:

A32016 SO2, BALN

Reference Number: 163-400807728-1 Cylinder Volume:

Cylinder Pressure: Valve Outlet:

Certification Date:

144.5 CF 2015 PSIG

660 Dec 21, 2016 ·

Expiration Date: Dec 21, 2024

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical Interference, This cylinder has a total analytical 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical Interference, This cylinder has a total analytical 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical Interference, This cylinder below 100 pstg. Let 0.07 megapasoals.

			Do Not Use This Cylinder b	elow too bard' i'e'	O.7 Miconpus	20101			
ANALYTICAL RESULTS Assay Assay									
Component		Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty		Dafes		
		1990 PPM	2001 PPM	G1 .	31 +/- 0;6% NIST Traceable		12/14/2016, 12/21/2016		
NITROGEN Balance :			· [
			CALIBRATI	ON STANI	DARDS	•	- 1		
Туре	Lot ID	Cylinder No	Concentration			Uncertainty	Explration Date		
NTRM	120105	KAL003384	2502 PPM SULFU	R DIOXIDE/NIT	ROGEN	+/-0.60%	Jun 04, 2019		
			ANALYTICAL EQUIPMENT Analytical Principle		Last Multipoint (Calibration .			
Instrument/Make/Model SO2-H - NIGOLET 6700 AHR0800411			FIR			Nov 28, 2016			
SO2-H - N	IGOLET 670	J AHRU500411	THE	The second secon	ىدە ئائىنىدەنلىكىنىدى <u>ت كىنىدىن</u>	والمستقدمة والمستحد والمستحد والمستحدد			

Triad Data Available Upon Request





Eco Services Operations Corp. Houston Plant

Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Unit #8

Stack O2 Analyzer

1st Quarter 2018 - RATA

RELATIVE ACCURACY TEST AUDIT REPORT ECO SERVICES OPERATIONS CORP. SULFURIC ACID UNIT NO. 8 HOUSTON, TEXAS TEST DATE: 14 MARCH 2018 RN 100220581/CN 605004464 PERMIT NO. 19282

Prepared for:

ECO SERVICES OPERATIONS CORP.

8715 Manchester Street Houston, TX 77012

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way West Chester, Pennsylvania 19380

April 2018

W.O. No. 15364.002.006

1. TEST PROGRAM SUMMARY

1.1 INTRODUCTION

The Eco Services Operations Corp. (Eco Services) Houston, TX facility is required to perform annual continuous emissions monitoring system (CEMS) Relative Accuracy Test Audits (RATAs) on its Sulfuric Acid Unit No. 8 (Unit 8) scrubber stack. This report provides a detailed description of the monitoring procedures that were used to demonstrate compliance for the sulfur dioxide (SO₂) and oxygen (O₂) CEMS.

Section 1 provides a summary of the overall test program and test parameters. Section 2 provides a summary of the Relative Accuracy Test Audit (RATA) results for each CEMS tested. A description of process operations, test locations, and sampling procedures are provided in Sections 3 through 5, respectively.

Appendices A through E provide Unit 8 CEMS and process operations data, reference method CEMS data, example calculations, quality control data, and project participants, respectively.

1.2 SAMPLE PROGRAM MATRIX

Table 1-1 provides a summary of test parameters and test procedures. All CEMS RATA testing was performed on 14 March 2018.

Table 1-1
Sample Program Matrix

Sample Parameter	Sample Location	Test Method	Number of Tests	Comments
Sulfur Dioxide	Stack CEMS	EPA Method 6C	11	ppmv and % relative accuracy
Oxygen	Stack CEMS	EPA Method 3A	11	% O ₂ and % difference

2. RESULTS AND DISCUSSION

The CEMS certification results are shown in Table 2-1. The test results for relative accuracy met the performance specification criteria for each CEMS tested. Eleven reference method test runs were conducted at the stack CEMS location. The relative accuracy was based upon the best nine of the eleven runs. Run No. 1 was not used in the RATA calculations due to a gas conditioner failure in the Weston CEM system. All eleven RATA test runs are reported in the appendices.

Appendix B provides detailed summaries of the relative accuracy and performance specification testing.

Table 2-1
Summary of CEMS Performance Specification Test Results
RN 100220581/CN 605004464, Permit No. 19282

	Relative Accuracy					
Stack Analyzers	Performance Required (%)	Performance Demonstrated (%)				
SO ₂	≤20	4.7				
O ₂	≤1	0.01				

3. DESCRIPTION OF PROCESS OPERATIONS

3.1 DESCRIPTION OF SULFURIC ACID UNIT NO. 8

The Eco Services Sulfuric Acid Unit No. 8 is a virgin sulfuric acid manufacturing unit. In the virgin sulfuric acid process, molten sulfur is sprayed into the furnace and burned at a high temperature with excess air to produce sulfur dioxide (SO₂); the SO₂ is cooled in a waste heat boiler. Gaseous SO₂ is reacted with air in a catalytic converter to produce sulfur trioxide (SO₃); between the first two stages of the converter, exothermically generated heat is removed by another waste heat boiler. The SO₃ is absorbed in strong acid in an absorbing tower to produce high-concentration sulfuric acid. Exhaust gas leaves the absorbing tower passing through an SO₂ scrubber before exiting from the stack.

4. SULFUR DIOXIDE CONTINUOUS EMISSIONS MONITORS (CEMS), LOCATIONS AND SPECIFICATIONS

Eco Services operates CEMS to measure SO_2 and O_2 concentrations at the scrubber stack. The CEMS specifications are:

Manufacturer	Serial Number	Range
	VE-920-8700-2	0-5000 ppm SO ₂
	VE-920-8700-2	0-25% O ₂
	Manufacturer Ametek 920 Ametek 920	Ametek 920 VE-920-8700-2



Eco Services Operations Corp. Houston Plant

Data Assessment Reports 40 CFR Part 60 Appendix F Procedure 1, Section 7

Unit #8

Stack O₂ Analyzer

2nd Quarter 2018 - CGA



Eco Services - Houston

Quarterly Cylinder Gas Audit Checklist U8 Stack O₂ Analyzer

Date: 5/25/2018

Time: 9:00AM

Serial Number: VE-920-8700-2

Technician: J. Thomas

Signature:

Revised: 05/28/18

Cylinder ID number	CC242908		DT0005743			
	8/30/2016		6/19/2017			
Date of Certification	EPA Pro. 1		EPA Pro. 1			-
Type of certification (e.g. EPA Protocol 1 or CRM).	EFA 110, 1					
	Trial 1		Tri	al 2	Trial 3	
	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2	Audit Point 1	Audit Point 2
Certified audit value C_a	5.00	15.01	5.00	15.01	5.00	15.01
(ppm) CEM Response value	4.98	15.20	5.02	15.20	5.01	15.20
C_m (ppm) Accuracy A (% or ppm)	-0.400%	1.266%	0.400%	1.266%	0.200%	1.266%

where $A = (C_{\underline{m}} - C_{\underline{n}}) \times 100$

COMPLIANCE CLASS

Guaranteed 1/- 2% Accuracy

9810 Bay Area Blvd., Pasadepa, TX 77507

Phone: 281-474-5800

Fax: 281-474-5950

29 10 DB) MED DIVER 1 BORDELLE IN THE	· · · · · · · · · · · · · · · · · · ·							
CERTIFICATE OF ACCURAC	Y : EPA Protocol (ias .					:	
Customer: SOLVAY USA INC SOLVAY USA INC - STOREROOM 8615 Manchester St Houston, TX 77012-2142 US	Assay Laboratory - PGVP Vendor ID: A32018 Air Liquide America Specially Gases LLC 9810 Bay Area Blvd. Pasadena, TX 77507				Lot No: 554861 P.O. No.: 4502361890 Folio #: 5% O2/N2 . Sales Order # : 5168220			
ANALYTICAL INFORMATION	Gas T	ype: O2,BALN				`6 d		
This certification was performed according EPA/600/R-12/531; May 2012. Do not use	lo EPA Traceability Protocol bis standard if pressure is le	For Assay & Certification ss than 100 psig.	n of Geseous Calibra					
Cylinder Number: CC242908		rtification Date: 3	0Aug2016		ation Date: o: 554861	`31Aug2'	024	
*		Concentratio (Mole)	on .		Accuracy	(Absolut	e / Relativ	/e)
Component Name OXYGEN NITROGEN		5,00 % BAI	ANCE		0.03	%	, 0,6	%
TRACEABILITY					· · · · · · · · · · · · · · · · · · ·			
Analytical Traceability Reference Standard Component OXYGEN ANALYTICAL METHOD	Concentration 20.8900 %	<u>Uncertainty</u> 0.11 %	<u>Cylindar</u> K014 168	Type NTRM	2659		Exp. D 27 Jun2	
AIDE HOLE		1st Analysis: 08/30	12016					
COMPONENT OXYGEN	<u>INSTRUMENT</u> SIEMENS OXYMAT DO	<u>AN.</u>	ALYTICAL PRINC amagnetic	<u>IPLE</u>	CALIBRATE 08/08/2016	<u>n co</u>	NCENTRA 5.00	ATION %

APPROVED BY: David Kelly

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Airgas USA, LLG 9810 BAY AREA BLVD Pasadens, TX 77507 Alrgas.com

CERTIFICATE OF ANALYSIS Grade of Product: EPA Protocol

Customer:

ECO SERVICES

OPERATIONS LLC

Part Number:

E02N185E15A3432

Cylinder Number:

DT0005743 124 - Pasadena (SG06) - TX

Laboratory: PGVP Number:

A32017

Gas Code:

O2,BALN

Reference Number: 163-400931411-1

Cylinder Volume:

145.7 CF 2015 PSIG

Cylinder Pressure: Valve Outlet:

590

Certification Date:

Jun 19, 2017

Expiration Date: Jun 19, 2025

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gassous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cythoder has a total analytical encounteral traceast the use of this calibration mixture. All concentrations are on a volume/volume basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals

Component	· Reques		ANALYTICAJ Actual Concentration	L RESU Protoco Method		Total Relative Uncertainty	Assay Dates
OXYGEN	15.00 %	ı ı	15,01 %!	G1		+/- 1,0% NIST Traceable	
NITROGEN Balance CALIBRATION STANDARDS							
Туре	Lot ID	Cylinder No	. Concentration			Uncertainty	Expiration Date
NTRM	103009	K021666 .	20,89 % OXYGEN			+/-0.63%	Jun 27, 2022
ANALYTICAL EQUIPMENT Instrument/Make/Model Analytical Principle Last Multipoint Calibration						Ibration	
	XYMAT 6 DD550)	PARAMAGNETIC		Jun 06, 2017		

Triad Data Available Upon Request



Approved for Release .!

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